APPENDICES

APPENDIX 31

1.0 OBJECTIVES

This Standard Operating Procedure (SOP) describes the procedures that the auditor will use for performing analytical laboratory audits and the reporting of the audit findings. Analytical laboratory audits will be performed to provide GE with an indication of the quality of the data that are being provided from the laboratories as part of the Hudson River Design Support Sediment Sampling and Analysis Program and to ensure that the laboratories are adhering to project requirements. This SOP applies to the laboratories and the auditor.

2.0 EQUIPMENT

Not applicable.

3.0 SUPPORTING SOPs and DOCUMENTS

Applicable Analytical SOPs.

Quality Assurance Project Plan (QAPP).

Applicable Field Sampling Plan (FSP).

Health and Safety Plan

Applicable Laboratory Quality Assurance Manual (LQAM)

4.0 PROCEDURE

4.1 SCHEDULING

The frequency of laboratory audits will be identified in the appropriate FSP and/or QAPP. When it has been determined that the performance of a laboratory audit is necessary, the auditor will initiate contact with the designated project laboratory. The auditor and the laboratory will mutually determine a date and time for the audit which is convenient for both parties and which

is within any deadline necessary to meet the needs of the FSP and/or QAPP. If necessary, unannounced audits may be performed.

4.2 PRE-AUDIT PREPARATION

At the time the laboratory audit is scheduled, the auditor will request any laboratory documents that would aid in the effectiveness of the audit if received prior to the audit. Such documents will include the LQAM, all analytical Standard Operating Procedures (SOPs) which are applicable to the analytical work being performed by the laboratory, and applicable recent Performance Evaluation (PE) sample analysis results.

Prior to the audit, the auditor will review all documents provided by the laboratory. In the laboratory audit report, a statement will be made as to the appropriateness and thoroughness of these documents in terms of the needs of the FSP and/or QAPP. In addition, the auditor will take notes upon this initial review that will aid in verifying that the laboratory is following the procedures described in the documents. Any deviations from the documents will also be noted in the laboratory audit report.

4.3 GENERAL AUDIT APPROACH

Upon arrival at the designated project laboratory, the auditor will initiate a meeting with the appropriate laboratory personnel (analytical laboratory supervisor(s) and quality assurance supervisor) to give a brief introduction of what they can expect to occur during the laboratory audit. This introduction focuses on several key points. The auditor(s) will identify that they will be performing the audit with the aid of a checklist that has been prepared by the auditor. The checklist will not be provided to the laboratory at any time.

This checklist guides the auditor(s) through the laboratory in the basic order that samples are processed through the laboratory starting with sample receipt and ending with data reporting. Another key point that will be made is that the auditor(s) will focus their questions toward the technicians and analysts who actually perform the work and **not** their supervisors. Laboratory supervisors may be present during the laboratory audit but may not answer the questions for the technicians/analysts unless the technicians/analysts cannot answer a given question. In addition, the auditor will ask questions regarding appropriate general laboratory information during this initial meeting.

The audit checklist is a proprietary document and has not been included with this SOP. It contains significant specific detail on the audit criteria and evaluation. The laboratory evaluation checklist includes the following eight sections. The laboratory audit will be performed in the basic order defined by the checklist, depending on the layout of the laboratory being audited. Subdivisions of the following sections will be necessary and will be dependent on the types of analytical work being performed for the FSP at the designated laboratory. The approach of the audit on the following topics is described in greater detail in the subsequent sections of this SOP. In addition to the following topics, the auditor(s) will also be evaluating the overall sample tracking throughout the audit and the communication between the various sections of the laboratory (with a special regard to how holding times are met). The narrative of the laboratory audit report will also follow the subject order of the checklist but will give the audit findings and recommendations in sufficient detail that the checklist will not be included with the report.

1. Organization and Personnel
2. Sample Receipt and Storage Area
3. Sample Preparation Area

4. Sample Analysis Instrumentation
5. Documentation
6. Quality Control Manual
7. Data Handling
8. Summary

Finally, following the audit, the auditor(s) will initiate another meeting with the appropriate laboratory personnel in order to debrief them on the audit findings. The debriefing meeting should accomplish several goals. First, the laboratory personnel must be made aware of the major points of the audit findings. This way, any statements made in the laboratory audit report will not be a surprise to the laboratory and the laboratory is given a chance to respond to the findings before the report is written. Their responses can then be incorporated into the laboratory audit report. Secondly, the auditor(s) should emphasize that recommendations will be made even to the best of laboratories. The major goal of a laboratory audit is to determine the quality of data which is currently being generated by the laboratory and to identify problems (or potential problems) so that appropriate corrective action can be initiated by the laboratory. However, laboratory audits are also attended to give all laboratories, no matter their level of quality, ideas on how to become a better laboratory. The extent of the impact of the recommendation(s) will be indicated in the report.

4.4 ORGANIZATION AND PERSONNEL

The auditor(s) must evaluate the size and experience of the organization and the number, working hours, experience, and education of the personnel in the context of the FSP analytical work which is being performed by the laboratory being audited. The general information about the organization and personnel should be addressed by the auditor(s) during the initial introduction meeting. The experience and education of the personnel are best evaluated at a later date by obtaining the staff's resumes during this introductory meeting. However, if the

resumes or some other similar summaries are not available, questions of this nature must be asked by the auditor(s) throughout the audit.

4.5 SAMPLE RECEIPT AND STORAGE AREA

The auditor(s) should interview the designated sample custodian in this area of the laboratory. If a sample custodian has not been designated by the laboratory, the auditor(s) must interview any personnel responsible for receiving and logging in samples upon receipt at the laboratory. The auditor(s) must determine whether the laboratory personnel do anything to compromise the integrity of the samples during the check-in process, such as subjecting samples to any procedures which might lead to contamination or allowing temperature-preserved samples to warm to room temperature. In addition, the auditor(s) must verify that the laboratory personnel check and record all appropriate information regarding the condition of the samples upon receipt (i.e., Chains-of-Custody, cooler temperature, preservation, etc.). Furthermore, the auditor(s) must determine whether the sample storage area(s) are kept at the proper well-documented temperatures without any possibility of cross-contamination of samples. Finally, the auditor(s) must verify that the laboratory properly documents the condition of the samples upon receipt and that the information is determined from the check-in procedure. It must also be determined whether the tracking system in which the samples have been logged is adequate to ensure that holding times are being met.

4.6 SAMPLE PREPARATION AREA

The auditor(s) must evaluate the overall appearance and appropriateness of the size of the sample preparation area and the condition of the facility and equipment in the sample preparation area. The auditor(s) must verify that the equipment, water, and standards used in sample preparation are appropriately calibrated, stored, and/or maintained and that all appropriate information regarding these issues is properly documented. While in this area, the auditor(s) must interview the sample preparation technicians responsible for the preparations for the analyses of concern for the FSP. During the interviews, the auditor(s) must determine whether the sample preparation technicians follow good laboratory practices as well as the required analytical preparation methods. The auditor(s) must evaluate whether the laboratory is introducing the appropriate type and number of quality control samples at this point and that the sample technicians' procedures or equipment do not introduce possible contamination (i.e., glassware is cleaned properly) or inaccuracies (i.e., proper glassware and standards are used correctly). The auditor(s) must also evaluate the laboratory's sample tracking system through this area to verify that holding times are properly tracked. Finally, the auditor(s) must verify whether all preparation procedures are appropriately documented.

4.7 SAMPLE ANALYSIS INSTRUMENTATION

In the various sample analysis instrumentation areas of the laboratory, the auditor(s) must interview the analyst(s) responsible for the analyses of concern for the FSP. When possible, analysts will also be observed performing assigned tasks. The auditor(s) must evaluate whether the instrumentation used for the analyses of concern for the FSP is appropriate and is properly maintained. Through the interviews with the analysts and observations of actual tasks, it must be determined if each instrument is calibrated (according to the associated analytical method) with well-documented calibration standards at the required frequency and that these instrument

calibrations are properly documented. The auditor(s) must determine if method detection limit studies have been performed on each instrument used for analysis. In addition, the auditor(s) must determine if standards and quality control samples are being analyzed at an appropriate frequency and whether appropriate quality control limits are being utilized for these analyses. The analysts' practices must be evaluated for their attention to the quality control results and to the possibility of instrument carryover. The auditor(s) must evaluate whether appropriate corrective actions are being taken when standard or quality control results are out of the method-required or laboratory-determined limits. Finally, the auditor(s) must determine whether all the analysis procedures and results are properly documented.

4.8 DOCUMENTATION

The auditor(s) must evaluate the laboratory's documentation procedures throughout the audit. It should be determined if well-labeled, neat, bound notebooks are being used to document and trace standards, calibrations, laboratory procedures, and any other routine quality control check. The auditor(s) must examine notebooks and observe whether they have been properly reviewed at the laboratory and that there are no obliterations.

The auditor(s) must also evaluate the data package preparation practices. Such practices include the system used for the collection of various hard copy data, validation of results prior to data package release, completeness checks, generation of cover letters or case narratives, and retention of data packages at the laboratory.

4.9 QUALITY CONTROL MANUAL

The appropriateness and thoroughness of the laboratory's quality control manual, as well as the LQAM and analytical SOPs, must be evaluated prior to the audit. However, throughout the audit, the auditor(s) must verify if the laboratory is actually following the practices defined in their documents. Any deviations from the documented procedures must be discussed in the laboratory audit report.

4.10 DATA HANDLING

The auditor(s) must determine whether the laboratory's data handling procedures are adequate. Proper data handling techniques include the checking of calculations by a second person, documenting calculations, recording all corrective actions taken on rejected data, and properly documenting detection limits and quality control results. In addition, data must be retained at the laboratory for an appropriate amount of time.

4.11 SUMMARY

The auditor(s) must summarize the major points of the laboratory audit findings and the overall impact on the quality of the data issued from the laboratory. The summary should also reflect the overall attitude that the laboratory has toward quality assurance and quality control.

4.12 REPORT FORMAT

The laboratory audit report must contain the following sections in the following order. It must also contain a completed checklist as an attachment.

1. Introduction
2. Executive Summary

3. Audit Findings

The introduction should summarize who performed the audit, when the audit was performed, the name and location of the laboratory, and the laboratory's role in the FSP. The Executive Summary should address the major findings of the laboratory audit along with the possible impact on the laboratory's data quality. The Audit Findings should follow the major headings of the checklist summarizing the findings presented on the checklist along with any recommendations for improvement or corrective action. The report will be signed by the auditor(s) who performed the audit.

5.0 TRAINING

The auditor's conducting the laboratory audit must have demonstrated knowledge in laboratory quality assurance/quality control practices. This knowledge will include experience in analytical data validation and having conducted at least two prior laboratory audits.

APPENDIX 32

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that Environmental Standards data reviewers will use to validate volatile organic data generated by SW-846 Method 8260B for the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to SW-846 Method 8260B and/or other reference documents (*e.g.*, analytical SOPs) as applicable to the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the volatile organic data provided by the analytical laboratory(ies) will be determined based on the general guidance provided in the "US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review" (10/99; National Functional Guidelines). It should be noted that the National Functional Guidelines applies strictly to data generated by Contract Laboratory Program (CLP) protocol and it is not directly applicable to validation of data generated by SW-846 Method 8260B; this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes, or a data summary spreadsheet of the qualified analytical results will be included in the analytical results section of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELForms:

- Organic field duplicate comparison Rev1-01.xls
- Organic field quadruplicate comparison Rev1-01.xls
- Organic field triplicate comparison Rev1-01.xls

Chemistry Applications:

- FIT
- Mass Spec Database
- Methods Database
- Target Version 4.1 data processing software

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/99).

- SW-846 Method 8260B.

- Region I, EPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, Standard Operating Procedure for the Validation of Organic Data Acquired Using SW-846 Method 8260B (Rev. 2, 12/96).

- Region III, Modifications to National Functional Guidelines for Organic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the volatile data based on an evaluation of information presented in the data package deliverables. Compliance with SW-846 Method 8260B and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of deficiencies prior to submittal of the QAR (if feasible and sanctioned by General Electric

Company). At a minimum, corrections required to allow for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the comments subsection of the QAR and will be included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the volatile organic data based on an evaluation of the information presented in the data package deliverables. The findings of the volatile organic data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data; these qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) in General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination, common contaminants that were not qualified, unusable results (R/UR), tentative identifications of target compounds (N), estimated results (J/UJ), tentatively identified compounds (TICs), field duplicate comparison, and a general qualifier for all results reported below the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the volatile organic data and the resultant qualifications will be as stated on the attached Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B. It should be noted that the Project Manager should be consulted when the use of "professional judgement" is indicated on the attached table.

Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B

Quality Control Item	Usability Criteria	Action(s)
Temperature and Conditions Upon Receipt (See Note #1 for additional information)	4±2°C. Aqueous samples should not have headspace.	If temperature is >6°C but ≤10°C, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If temperature is >10°C, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If aqueous samples are received with headspace >pea-size but ≤5 mL, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If aqueous samples are received with headspace >5mL, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Note the time of sample collection relative to receipt at the laboratory; use professional judgement if < 8 hours has elapsed from collect to receipt to determine if the qualification for elevated temperature applies.
Technical Holding Time (See Note #2 for additional information)	Chemically preserved (pH≤2 with HCl) aqueous samples analyzed for aromatic compounds should be analyzed within 14 days of collection. Preserved solid/soil samples should be analyzed within 14 days of collection. Unpreserved (pH>2) aqueous samples analyzed for aromatic compounds should be analyzed within 7 days of collection.	If holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If holding time is grossly exceeded (*i.e.,* >twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
GC/MS Tuning (BFB) (See Note #3 for additional information)	Ion abundances should meet the method acceptance criteria.	If mass calibration was not performed, qualify all associated data as unusable ("R/UR"). If mass assignment is in error, qualify all associated data as unusable ("R/UR"). Use professional judgement if abundance criteria are not met. (See Note #3.) Use professional judgement if samples are analyzed more than 12 hours after a compliant tune and there is no evidence of a compliant tune following the samples.

Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B

Quality Control Item	Usability Criteria	Action(s)
Initial Calibration (See Note #4 for additional information.)	Average relative response factor (RRF) for each compound should be ≥0.050. %RSD should be ≤15% or a calibration curve should be generated. If a curve is generated, r (linear) or coefficient determination (COD; quadratic) should be ≥0.99.	If target compounds have an average RRF <0.050, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If target compounds have 15%< %RSD ≤50%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If target compounds have 50%< %RSD ≤90%, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. If target compounds have %RSD > 90%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Use professional judgement when evaluating correlation coefficients (r) and coefficients of determination (COD). If r (linear) or COD (quadratic) is <0.99 but ≥0.85, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If r or COD is <0.85, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the initial calibration standards and the samples associated were not performed similarly (*e.g.,* the initial calibration standards were heated and the samples were not heated), qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B

Quality Control Item	Usability Criteria	Action(s)
Continuing Calibration Verification (CCV) (See Note #5 for additional information.)	CCV RRFs for target compounds should be ≥0.050. %drift or % difference (%D) should be ≤20%.	If target compounds have an RRF <0.050, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If target compounds have 20%< %D ≤90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If target compounds have %D>20% with a response indicating a sensitivity increase, qualify positive results as estimated ("J") and use professional judgment to qualify "not-detected" results. If target compounds have %D>90%, with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Internal Standards	Area counts of the internal standard peaks should be 50-200% of the internal standard area observed in the associated CCV standard. Retention time (RT) for any internal standard should not vary by more than ±30 seconds from RT in the associated CCV standard.	If a sample area count is outside of criteria (50-200% of associated CCV), qualify positive results for compounds quantitated using that internal standard as estimated ("J") and qualify "not-detected" results for compounds quantitated using that internal standard as estimated ("UJ"). If extremely low sample area counts (<25%) are reported, qualify positive results for compounds quantitated using the extremely low internal standard as estimated ("J") and qualify "not-detected" results for compounds quantitated using that internal standard as unusable ("UR"). If an internal standard RT varies by more than 30 seconds and no peaks are observed in the sample chromatogram, qualification is not necessary. Use professional judgement if peaks are observed in the sample chromatogram.

Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B

Quality Control Item	Usability Criteria	Action(s)
Blanks (See Notes #6 and for additional information.)	Summarize all results greater than the method detection limit (MDL) in the blanks. The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a target compound is detected in the blank but not in the associated sample(s), no action is required. If a sample result is ≤5× (10× for common contaminants) blank result, qualify the positive result as "not-detected" ("U*"). If the positive result qualified "U*" is <RL, the RL should be used as reported. If the positive result qualified "U*" is ≥RL, the value of the positive result should be used as the revised RL. If a sample result is >5× (10×) the blank result, qualification is not required. If gross contamination exists (*i.e.*, saturated peaks by GC/MS), qualify the positive results as unusable ("R") due to interference. If a TIC is observed in blank and sample, or if the TIC is a known laboratory artifact, qualify the TIC result as unusable ("R").
Surrogate Recovery	Use laboratory acceptance limits. Use 70%-130% as the default limits if the laboratory limits are unreasonable.	If recoveries of one or more surrogates > upper limit, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If recoveries of one or more surrogates < lower limit, but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If recoveries of one or more surrogates <10%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B

Quality Control Item	Usability Criteria	Action(s)
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	For accuracy use recovery limits of 70-130%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if the original concentration of a compound is >4× the spiking level for that compound. RPDs calculated using MS/MSD results can be used to evaluate precision. If the recovery is >130%, qualify the positive result in the native sample as estimated ("J") and do not qualify the "not-detected" result. If the recovery is <70% but ≥30%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as estimated ("UJ"). If the recovery is <30%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive result in the native sample as estimated ("J") and do not qualify the "not-detected" result. If the precision criteria (see field duplicate usability criteria) for non-spiked compounds are not met, qualify positive results in the native sample as estimated ("J") and qualify "not-detected" results in the native sample as estimated ("UJ"). If a field duplicate of the native sample was collected and analyzed, the field duplicate should also be qualified if the MS/MSD %Rs or RPD are outside of the criteria (as stated above for the native sample).
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	For accuracy, use recovery limits of 70-130%. For precision, use RPD limit of 20% for aqueous samples and 40% for solid samples.	If the recovery is >130%, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery is <70% but ≥30%, qualify positive results in the associated samples as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recovery is <30%, qualify positive results in the associated samples as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the precision exceeds the RPD criterion, qualify positive results in the associated samples as estimated ("J") and do not qualify "not-detected" results.

Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B

Quality Control Item	Usability Criteria	Action(s)
Field Duplicate (See Note #7 for additional information.)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are ≥5× RL. Use limit of ± RL (±2× RL for solids) when at least one sample value is <5× RL. (Use one-half the RL as a numerical value for any "not-detected" results in the RPD calculations).	If the criteria are not met, qualify positive results for non-compliant compounds in original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Target Compound Identification (See Note #8 for additional information.)	Relative Retention Time (RRTs) should be ±0.06 RRT units of the standard RRT. Mass spectra of sample and current laboratory-generated standard should match.	Use professional judgement when applying the qualitative criteria for GC/MS analysis of target compounds. If it is determined that incorrect identifications were made or positive results were not reported, professional judgment should be used to determine which of the following options should be used: (1) qualify affected results as unusable ("R"); (2) correct reported results based on the raw data; or (3) contact laboratory for clarification. If a positive result meets some qualitative criteria but an evaluation of all qualitative criteria is inconclusive (possibly due to mass spectral interferences), use professional judgment to determine if result should be qualified "N".
Percent Solids	Solid samples with less than 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.

Table for the Validation of Volatile Organic Compounds Generated by SW-846 Method 8260B

Quality Control Item	Usability Criteria	Action(s)
Compound Quantitation (See Note #9 for additional information.)	Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution.	If target compound results exceeds the instrument calibration range, qualify positive results as estimated ("J"). If a target compound result is <RL but ≥MDL, qualify positive results as estimated ("J"). Use professional judgment to determine whether sample reanalyses and dilutions should be compared to the original analysis. If the precision criteria (see field duplicate usability) between the original sample results and the reanalysis sample result are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
System Performance (See Note #10 for additional information.)	Professional judgment should be used when assessing the degradation of the system performance during analyses.	Use professional judgement to qualify the data if it is determined that system performance degraded during sample analysis.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data that were not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitation of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

Notes for the Validation of the Volatile Organic Data Generated by SW-846 Method 8260B

1. If several sample vials are received at the laboratory for one aqueous sample and the laboratory indicates that only one vial was received with head space, the data should not be qualified. It may be necessary to contact the laboratory to determine if the laboratory utilized a sample vial without head space.

2. If the pH is not checked by the laboratory, contact the Project Manager to determine if pH logs should be requested from the laboratory. If pH logs are not obtained, check the chain-of-custody (COC) to determine if the sample was properly preserved. Assume the pH is within the specified criteria if the COC indicates the sample was preserved and include a comment in the QAR indicating this.

   If a sample is acid-preserved (HCl for aqueous or sodium bisulfate for solids), the priority pollutant level (PPL) compound 2-chloroethyl vinyl ether may not be recovered because it is an acid-labile compound. This lack of recovery can be confirmed by examining the MS/MSD %Rs of an acid-preserved sample. In acid-preserved samples, qualify positive results for 2-chloroethyl vinyl ether as estimated ("J") and qualify "not-detected" results for 2-chloroethyl vinyl ether as unusable ("R").

3. Mass Tuning Criteria (alternate tuning criteria may be used by laboratory [*e.g.*, CLP, Method 524.2, or manufacturer instructions] provided that method performance is not adversely affected.)

Notes for the Validation of the Volatile Organic Data Generated by SW-846 Method 8260B

Mass	Intensity Required
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

If using professional judgement to determine an impact when ion abundance criteria are not met, the most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of less importance.

4. If the initial calibration %RSD is >50%, the linearity of the first three initial calibration standards for the compound should be evaluated. If the first three initial calibration standards for the compound are linear (*i.e.*, r ≥0.99), do not qualify "not-detected" results. If the first three initial calibration standards for the compound are not linear, qualify "not-detected' results as estimated ("UJ").

Notes for the Validation of the Volatile Organic Data Generated by SW-846 Method 8260B

Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive, the samples should be evaluated for false positives. If the concentration intercept is negative, the samples should be evaluated for false negatives.

5. If instrument instability (*i.e.*, several continuing calibration standards with compounds exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential continuing calibration standards, "not-detected" results may be qualified as estimated ("UJ") due to instrument sensitivity of a continuing calibration standard response that is greater than the initial calibration standard response (increase in instrument sensitivity).

   If the continuing calibration standard is %D>20% in the direction of increased instrument sensitivity and it is determined that "not-detected" results should not be qualified, the data reviewer should note this in the QAR support documentation.

6. The frequency of equipment/rinse/storage blanks is determined during the sampling event. The results of a field/equipment/rinse blank should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day; unless only one was collected for a several-day sampling event. In instances when more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration for a contaminant.

Notes for the Validation of the Volatile Organic Data Generated by SW-846 Method 8260B

The following compounds are considered common laboratory contaminants/artifacts. If these compounds are reported as target compounds, the 10× rule applies for the evaluation of blank contamination. If these compounds are reported as TICs, they should be considered laboratory artifacts.

a. Common laboratory contaminants on CLP target compound list (TCL): methylene chloride, acetone, 2-butanone, and cyclohexane.

b Other common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, freons, and phthalates.

c. Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexanone, cyclohexanol, chlorocyclohexene, and chlorocyclohexanol.

d. Aldol condensation reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

e. Silicon-containing compounds (*e.g.*, trimethyl silonol).

If a sample result qualified "U*" is < the RL and the laboratory did not report the RL on the data tables or Form the positive result (*e.g.*, 8 µg/L) should be replaced with the RL (*e.g.*, 10 µg/L).

7. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. It is also expected that soil duplicate results will have a greater variance

Notes for the Validation of the Volatile Organic Data Generated by SW-846 Method 8260B

than aqueous duplicate results.

8. Generally, all ions present in standard mass spectrum at relative intensity >10% should be present in the sample mass spectrum; however, the concentration of the compound should be considered when evaluating ions with a relative intensity of ≤30% in the standard mass spectrum. In addition, data for ions with a mz of ≤40 amu are often not collected and generally are not used for evaluation purposes.

   Characteristic ions from the reference mass spectrum (three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum) should maximize in the same scan or within one scan of each other. Relative intensities of these ions should agree within ±30% between the standard and the sample spectra.

   Ions that are not present in the standard mass spectrum may be present in the sample mass spectrum without impacting the qualitative identification of the target compound. The presence of such ions at relative intensity 10% may be indicative of a coeluting compound. If feasible, the coeluting compound should be tentatively identified in order to evaluate whether the coeluting compound's mass spectrum contains any of the characteristic ions of the target compound. If a coeluting compound that has a mass spectrum containing characteristic ions of the target compound is tentatively identified, use professional judgment to evaluate the impact on the sample result.

Notes for the Validation of the Volatile Organic Data Generated by SW-846 Method 8260B

9. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

10. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

- High background levels or shifts in absolute RT of internal standards
- Excessive baseline rise at elevated temperatures
- Extraneous peaks
- Loss of resolution
- Peak failing or peak splitting that may result in an inaccurate quantitation

11. The RL will be defined on a project-specific basis. If the project-required RL is lower than the low initial calibration standard concentration, the Project Manager should be consulted for instructions on how to apply qualification related to the RL.

APPENDIX 33

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that Environmental Standards data reviewers will use to validate semivolatile organic data generated by SW-846 Method 8270C for the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to SW-846 Method 8270C and/or other reference documents (*e.g.*, analytical SOPs), as applicable to the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the semivolatile organic data provided by the analytical laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review" (10/99; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by Contract Laboratory Program (CLP) protocol and it is not directly applicable to validation of data generated by SW-846 Method 8270C; this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes, or a data summary spreadsheet of the qualified analytical results will be included in the analytical results section of the QAR.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELforms:

- Organic field duplicate comparison Rev 1-01.xls
- Organic field quadruplicate comparison Rev 1-01.xls
- Organic field triplicate comparison Rev 1-01.xls

Chemistry Applications:

- FIT
- Mass Spec Database
- Methods Database
- Target Version 4.1 data processing software

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/99).

- SW-846 Method 8270C.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

- Region I, EPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, EPA – SOP for Data Validation for SW-846 Method 8270C.

- Region III – Modifications to National Functional Guidelines for Organic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the semivolatile data based on evaluation of information presented in the data package deliverables. Compliance with SW-846 Method 8270C and/or other reference documents (*e.g.*, analytical SOPs or QAPjPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate

any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratory(ies) to request the correction of deficiencies prior to submittal of the QAR (if feasible and sanctioned by General Electric Company). At a minimum, corrections required to allow for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the comments subsection of the QAR and will be included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the semivolatile organic data based on an evaluation of the information presented in the data package deliverables. The findings of the semivolatile organic data usability assessment will be presented in terms data qualifications that the project team should consider in order to best utilize the data; these qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) in the General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

order: blank qualification, common contaminants that were not qualified, unusable results (R/UR), tentative identifications of targets (N), estimated results (J/UJ), tentatively identified compounds (TICs), field duplicate comparison, and a general qualifier for all results reported below the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the semivolatile organic data and the resulting qualifications will be as stated on the attached Table for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C. It should be noted that the Project Manager should be consulted when the use of "professional judgement" is indicated on the attached table.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	4±2°C.	If temperature is >6°C but ≤10°C, no action is required. If temperature is >10°C but ≤20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If temperature is >20°C, qualify positive results as estimated ("J") and qualify "not-detected" results unusable ("UR"). Note time of sample collection relative to receipt at laboratory; use professional judgment if < 8 hours has elapsed from collection to receipt to determine if the qualification for elevated temperature applies.
Technical Holding Time	Aqueous samples should be extracted within 7 days of sample collection. Solid/soil samples should be extracted within 14 days of sample collection. All matrices should be analyzed within 40 days after extraction.	If a holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a holding time is grossly exceeded (*i.e.*, >twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
GC/MS Tuning (DFTPP) (See Note #1 for additional information.)	Ion abundances should meet the method acceptance criteria.	If mass calibration was not performed, qualify all associated data as unusable ("R"/"UR"). If mass assignment is in error, qualify all associated data as unusable ("R"/"UR"). Use professional judgement if abundance criteria are not met. (See Note #1.) Use professional judgement if samples are analyzed more than 12 hours after a compliant tune and there is no evidence of a compliant tune following the samples.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Initial Calibration (See Note #2 for additional information.)	Average RRF for each compound should be ≥0.050. %RSD should be ≤15% or a calibration curve should be generated. If a curve is generated, r (linear) or coefficient of determination (COD; quadratic) should be ≥0.99.	If a target compound has an average RRF <0.050, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Compounds with 15%<%RSD≤50%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. Compounds with 50%< %RSD ≤ 90%, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. Compounds with %RSD > 90%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Use professional judgment when evaluating correlation coefficients (r) and coefficients of determination (COD). If r (linear) or COD (quadratic) is <0.99 but ≥0.85, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If r or COD is <0.85, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Continuing Calibration Verification (CCV) (See Note #3 for additional information.)	CCV RRFs for target compounds should be ≥0.050. %drift or %difference should be ≤20%.	If target compounds have an RRF<0.050, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If target compounds have 20%<%D≤90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If target compounds have %D>20% with a response indicating a sensitivity increase, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. If target compounds have %D>90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Internal Standards	Area counts of the internal standard peaks should be 50-200% of the internal standard area observed in the associated CCV. Retention time (RT) of the internal standard should not vary more than ±30 seconds from the RT of the internal standards observed in associated CCV standard.	If a sample area count is outside of the criteria (50-200% of associated CCV), qualify positive results for compounds quantitated using that internal standard as estimated ("J") and qualify "not-detected" results for compounds quantitated using that internal standard as estimated ("UJ"). If extremely low sample area counts (<25%) are reported, qualify positive results for compounds quantitated using that internal standard as estimated ("J") and qualify "not-detected" results for compounds quantitated using that internal standard as unusable ("UR"). If an internal standard RT varies by more than 30 seconds and no peaks are observed in the sample chromatogram, qualification of data is not necessary. Use professional judgment if peaks are observed in the sample chromatogram.
Blanks (See Notes #4 and #10 for additional information)	Summarize all results greater than the method detection limit (MDL) in the blanks. The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a target compound is detected in the blank but not in the associated sample(s), no action is required. If a sample result is ≤5× (10× for common contaminants) blank result, qualify the positive result as "not-detected" ("U*"). If the positive result qualified "U*" is <RL, the RL should be used as reported. If the positive result qualified "U" is ≥RL, the value of the positive result should be used as the revised RL. If a sample result is >5× (or 10×) blank result, qualification is not required. If gross contamination exists (*i.e.,* saturated peaks by GC/MS), qualify the positive results as unusable ("R") due to interference. If a TIC is observed in blank and sample or if the TIC is a known laboratory artifact, qualify as unusable ("R") due to interference.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Surrogate Recovery (See Note #5 for additional information.)	Use laboratory acceptance limits. Use default limits of 20-150% if laboratory limits are unreasonable.	If the recoveries of one or more surrogate in either fraction (acid or base) are > upper limit, qualify positive results for that fraction as estimated ("J") and do not qualify "not-detected" results. If the recoveries of one or more surrogate in either fraction (acid or base) are < lower limit but ≥10%, qualify positive results for that fraction as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recoveries of one or more surrogate in either fraction (acid or base) are < 10%, qualify positive results for that fraction as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Use 50-135% as recovery limits. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if the original concentration of a compound is > 4× spiking level for that compound. RPDs calculated using MS/MSD results can be used to evaluate precision. If the recovery is > 135%, qualify the positive result in the native sample as estimated ("J") and do not qualify the "not-detected" result. If the recovery is <50% but ≥10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as estimated ("UJ"). If the recovery is <10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native samples as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive result in the native sample as estimated ("J") and do not qualify "not-detected" result. If the precision criteria (see field duplicate usability criteria) for non-spiked compounds are not met, qualify positive results in the native sample as estimated ("J") and qualify "not-detected" results in the native sample as estimated ("UJ"). If a field duplicate of the native sample was collected and analyzed, the field duplicate should also be qualified if the MS/MSD %Rs or RPD are outside of the criteria (as stated above for the native sample).

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	Use 50-135% as recovery limits. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	If the recovery is >135%, qualify all positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery is <50% but ≥10%, qualify all positive results in all associated samples as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recovery is <10%, qualify all positive results in all associated samples as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the precision exceeds the RPD criterion, qualify positive results as estimated ("J") and do not qualify "not-detected" results.
Field Duplicate (See Note #6 for additional information.)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are ≥5× RL. Use limit of ± RL (±2× RL for solids) when at least one sample value is <5× RL. (Use one-half the RL as a numerical value for any "not-detected" results in the RPD calculations).	If the criteria are not met, qualify positive results for noncompliant compounds in original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Target Compound Identification (See Note #7 for additional information.)	Relative Retention Time (RRT) should be ±0.06 RRT units of the standard RRT. Mass spectra of sample and current laboratory-generated standard should match.	Use professional judgement when applying the qualitative criteria for GC/MS analysis of target compounds. If it is determined that incorrect identifications were made or positive results were not reported, professional judgement should be used to determine which of the following options should be used: (1) qualify affected results as unusable ("R"); (2) correct reported results based on the raw data; or (3) contact laboratory for clarification. If a positive result meets some qualitative criteria but an evaluation of all qualitative criteria is inconclusive (possibly due to mass spectral interferences), use professional judgment to determine if result should be qualified "N".
Percent Solids	Solid samples with less than 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.
Compound Quantitation (See Note #8 for additional information.)	Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution.	If a target compound result exceeds the instrument calibration range, qualify the positive result as estimated ("J"). If a target compound result is < RL but ≥ MDL, qualify positive results as estimated ("J"). Use professional judgement to determine whether sample reanalyses and dilutions should be compared to the original analyses. If the precision criteria (see field duplicate usability) between the original sample result and the reanalysis sample result are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a target compound result is <RL but ≥MDL, qualify positive results as estimated ("J").
System Performance (See Note #9 for additional information.)	Professional judgement should be used when assessing the degradation of the system performance during analyses.	Use professional judgement to qualify the data if it is determined that system performance degraded during sample analysis.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

Quality Control Item	Usability Criteria	Action
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgment to determine the need to qualify data not qualified based on the QC previously addressed. Write a brief narrative to give the user an indication of the analytical limitation of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

1. GC/MS Tuning Criteria (Alternate tuning criteria may be used by the laboratory [*e.g.*, CLP, Methods 525, or manufacturer instructions], provided that method performance is not adversely affected.)

m/z	ion abundance criteria
51	30-60% of m/z 198
68	less than 2% of m/z 69
70	less than 2% of m/z 69
127	40-60% of m/z 198
197	less than 1% of mass 198
198	base peak, 100% relative abundance
199	5-9% of m/z 198
275	10-30% of m/z 198
365	greater than 1% of m/z 198
441	present, but less than m/z 443
442	greater than 40% of m/z 198
443	17-23% of m/z 442

If using professional judgement to determine impact when ion abundance criteria are not met, some of the most critical factors in the DFTPP criteria are the non-instrument specific requirements that are also unduly affected by the location of the spectrum on the chromatographic profile. The m/z ratios for 198/199 and 442/443 are critical. These ratios are based on the natural abundances of C^{12} and C^{13} and should always be met. Similarly, the relative abundances for m/z 68, 70, 197, and 441 indicate the condition of

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

the instrument and the suitability of the resolution adjustment and are very important. For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. The relative abundance of m/z 365 is an indicator of suitable instrument zero adjustment. If relative abundance of m/z 365 is zero, MDLs may be affected.

2. If the initial calibration %RSD is >50%, the linearity of the first three initial calibration standards for the compound should be evaluated. If the first three initial calibration standards for the compound are linear (*i.e.*, r ≥0.99), do not qualify "not-detected" results. If the first three initial calibration standards for the compound are not linear, qualify "not-detected" results as estimated ("UJ").

   Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive, the samples should be evaluated for false positives. If the concentration intercept is negative, the samples should be evaluated for false negatives.

3. If instrument instability (*i.e.*, several continuing calibration standards with compounds exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential continuing calibration standards, "not-detected" results may be qualified as estimated ("UJ") due to instrument sensitivity of a continuing calibration standard response that is greater than the initial calibration standard response (increase in instrument sensitivity).

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

If the continuing calibration standard is %D>15% in the direction of increased instrument sensitivity and it is determined that "not-detected" results should not be qualified, the data reviewer should note this in the QAR support documentation.

4. The frequency of equipment/rinse blanks is determined during the sampling event. The results of a equipment/rinse blank should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day; unless, if only one was collected for a several-day sampling event. In instances when more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration for a contaminant.

   The following compounds are considered common laboratory contaminants/artifacts. If these compounds are reported as target compounds, the 10× rule applies for the evaluation of blank contamination. If these compounds are reported as TICs, the compounds should be considered laboratory artifacts.

   - Common phthalate contaminants.
   - Other common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, and certain freons.
   - Solvent preservatives, such as cyclohexene, which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanone.

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

- Aldol reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.
- Silicon containing compounds (*e.g.*, trimethylsilanol)

If a sample result qualified "U*" is <RL and the laboratory did not report the RL on the data tables or Form I the positive result (*e.g.*, 8 µg/L) should be replaced with the RL (*e.g.*, 10 µg/L).

5. The surrogate recovery limits do not apply to samples analyzed at dilutions greater than five-fold. Qualification of the data is not necessary if the surrogate is diluted beyond detection. Generally, greater than a five-fold dilution will affect the ability to even detect the surrogate. If a sample was analyzed at a five-fold dilution or less and the surrogates were not detected in the sample, qualify positive results as estimated ("J") and "not-detected" results estimated ("UJ").

6. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. It is also expected that soil duplicate results will have a greater variance than aqueous duplicate results.

7. Generally, all ions present in the standard mass spectrum at relative intensity >10% should be present in the sample mass spectrum; however, the concentration of the

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

compound should be considered when evaluating ions with a relative intensity of ≤30% in the standard mass spectrum. In addition, data for ions with a m/z of ≤40 are often not collected and generally are not used for evaluation purposes.

Characteristic ions from reference mass spectrum (three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum) should maximize in the same scan or within one scan of each other. Relative intensities of these characteristic ions should be within ±30% between the standard and sample spectra.

Ions that are not present in the standard mass spectrum may be present in the sample mass spectrum without impacting the qualitative identification of the target compound. The presence of such ions at relative intensity greater than 10% may be indicative of a coeluting compound. If feasible, the coeluting compound should be tentatively identified in order to evaluate whether the coeluting compound's mass spectrum contains any of the characteristic ions of the target compound. If a coeluting compound that has a mass spectrum containing the characteristic ions of the target compound, use professional judgment to evaluate the impact on the sample result is tentatively identified.

N-nitrosodiphenylamine (TCL, PPL, App IX) cannot be distinguished from diphenylamine (App IX) because *N*-nitrosodiphenylamine degrades to diphenylamine in the injection port. A note to this effect should be included in the qualifier section if these compounds are detected. In addition, benzo(b)fluoranthene and benzo(k)fluoranthene

Notes for the Validation of Semivolatile Organic Data Generated by SW-846 Method 8270C

sometimes coelute and have similar mass spectra; therefore, if either of these compounds is detected in samples, the chromatograms of the samples and standards should be evaluated for separation. If coelution is observed, qualify "J" and/or "N" using professional judgment.

8. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis to a diluted reanalysis.

9. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

- high background levels or shifts in absolute retention times of internal standards
- excessive baseline rise at elevated temperatures
- extraneous peaks
- loss of resolution
- peak tailing or peak splitting that may result in inaccurate quantitation

10. The RL will be defined on a project-specific basis. If the project-required RL is lower than the low initial calibration standard concentration, the Project Manager should be consulted for instructions on how to apply qualification related to the RL.

APPENDIX 34

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate organochloride pesticide data generated by SW-846 Method 8081A for General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to SW-846 Method 8081A and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the pesticide data provided by the analytical laboratory(ies) will be determined based on the general guidance provided in the "US EPA Contract Laboratory National Functional Guidelines for Organic Data Review" (10/99; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by SW-846 Method 8081A; therefore, this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes or a data summary spreadsheet of the qualified analytical results, will be included in the support documentation of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELforms:

- Organic field duplicate comparisons Rev1-01.xls
- Organic field quadruplicate comparison Rev1-01.xls
- Organic field triplicate comparison Rev1-01.xls

Chemistry Applications:

- FIT
- Methods Database
- Target Version 4.1 data processing software

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/99).

- SW-846 Method 8081A.

- Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, Standard Operating Procedure for the Validation of Organic Data Acquired Using SW-846 Method 8081A (Rev 2. 12/96).

- Region III, Modifications to National Functional Guidelines for Organic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the pesticide data based on an evaluation of information presented in the data package deliverables. Compliance to SW-846 Method 8081A and/or other reference documents (*e.g.,* analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of certain deficiencies prior to submittal of the QAR (if feasible and sanctioned by General Electric Company). At a minimum, corrections required to allow for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a

significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the comments subdivision of the QAR and included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the pesticide data based on an evaluation of the information presented in the data package deliverables. The findings of the pesticide data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data; these qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) in the General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination, unusable (R/UR) results, tentative identifications of target compound results (N), estimated (J/UJ) results, field duplicate comparison, and a general qualifier for all results reported below the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the pesticide data and the resultant qualifications will be as stipulated on the attached Table for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A. It should be noted

that the Project Manager should be consulted when "professional judgement" use is indicated on the attached table.

Table for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	4±2°C	If temperature is >6° but ≤10°C, no action is required. If temperature is > 10°C but ≤20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If temperature is >20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Note time of collection relative to receipt at laboratory; use professional judgement if < 8 hours has elapsed from collection to receipt at the laboratory to determine if qualification due to elevated temperature applies.
Technical Holding Time	Aqueous samples should be extracted within 7 days after sample collection. Solid/soil samples should be extracted within 14 days after sample collection. All matrices should be analyzed within 40 days after extraction.	If a holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a holding time is grossly exceeded (*i.e.*, > twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
GC Instrument Performance (See Note #1 for additional information.)	% Breakdown for 4,4′-DDT and endrin should be ≤15% for both GC columns.	Use professional judgement to determine if the associated sample data should be qualified if the instrument performance standard was not analyzed at the proper frequency. See Note #1 for action if the instrument performance standard criteria are not met.

Table for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Quality Control Item	Usability Criteria	Action
Initial Calibration (See Note #2 for additional information.)	%RSD should be ≤20% or a calibration curve should be generated. If a curve is generated, the curve should have r (linear) or coefficient of determination (COD; quadratic) must be ≥0.99.	If target compounds have 20%<%RSD≤50%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If target compounds have 50%<%RSD≤90%, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. If target compounds have %RSD>90%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Use professional judgement when evaluating correlation coefficients (r) and coefficients of determination (COD). If r (linear) or COD (quadratic) is <0.99 but ≥0.85, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If r or COD is <0.85, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Continuing Calibration Verification (CCV) (See Note #3 for additional information.)	%drift or %difference (%D) should be ≤15%.	Qualification is for all samples on both sides of the out-of-criteria calibration standards. If target compounds have 15%<%D≤90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If target compounds have %D>15% with the response indicating a sensitivity increase, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. If target compounds have %D>90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Quality Control Item	Usability Criteria	Action
Retention Time Windows (See Note #4 for additional information.)	All target compound retention times (RTs) should be within the established RT windows. RT windows should be estimated or defined by the laboratory as 3× the standard deviation of three non-sequential standards over a 72-hour period.	If the CCV RT windows are not within the specific RT windows, evaluate the chromatograms for false positives and false negatives. If a constant drift in RT is observed in the bracketing CCVs, the direction of the RT drift should be applied to the sample chromatograms.
Blanks (See Note #5 and Note #10 for additional information.)	Summarize all results greater than the method detection limit (MDL) present in the blanks. The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a target compound is found in the blank but not in the associated sample(s), no action is required. If a sample result is ≤5x the blank result, qualify the positive result as "not detected" ("U*"). If the positive result qualified "U*" is <RL, the RL should be used as reported. If the positive result qualified "U*" is ≥RL, the value of the positive result should be used as the revised RL. If a sample result is >5× the blank result, qualification is not required. If gross contamination exists (*i.e.*, saturated peaks on both GC columns), qualify the positive results as unusable ("R") due to interference.
Internal Standards (if used)	Area counts of the internal standard peaks should be 50-200% of the internal standard area observed in the associated CCV standard. RT for any internal standard should not vary by more than ±30 seconds from RT in the associated CCV standard.	If a sample area count is outside of criteria (50-200%), qualify positive results for compounds quantitated using that internal standard as estimated ("J") and qualify "not-detected" results for compounds quantitated using that internal standard as estimated ("UJ"). If extremely low sample area counts (<25%) are reported, qualify positive results for compounds quantitated using the extremely low internal standard as estimated ("J") and qualify "not-detected" results for compounds quantitated using that internal standard as unusable ("UR"). If an internal standard RT varies by more than 30 seconds and no peaks are observed in the sample chromatogram, qualification is not necessary. Use professional judgement if peaks are observed in the sample chromatogram.

Table for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Quality Control Item	Usability Criteria	Action
Surrogates (See Note #6 for additional information.)	Use laboratory acceptance limits. Use 20-150% as a default limit if the laboratory limits are unreasonable.	If samples are analyzed on two columns, samples should only be qualified if the out-of-criteria surrogate result is reported from the same column as the sample results. If the recoveries of one or more surrogates are > upper limit, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the recoveries of one or more surrogates are < lower limit but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recoveries of one or more surrogates are <10%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	For accuracy, use recovery limits of 50-135%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if the original concentration of a compound is 4× the spiking level for that compound. RPDs calculated using MS/MSD result can be used to evaluate precision. If the recovery is > 135%, qualify the positive result in the native sample as estimated ("J") and do not qualify "not-detected" results. If the recovery is < 50% but ≥10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as estimated ("UJ"). If the recovery is <10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive result in the native sample as estimated ("J") and do not qualify "not-detected" result. If the precision criteria (see field duplicate usability criteria) are not met for non-spiked compounds, qualify the positive result as estimated ("J") and qualify the "not-detected" result as estimated ("UJ"). If a field duplicate of the native sample was collected and analyzed, the field duplicate sample should also be qualified if an MS/MSD recovery or RPD is outside of criteria (as stated above for the native sample).

Table for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Quality Control Item	Usability Criteria	Action
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	For accuracy, use recovery limits of 50-135%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	If the recovery > 135%, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery < 50% but ≥10%, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as estimated ("UJ"). If the recovery is <10%, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as unusable ("UR"). If the precision exceeds the RPD criterion, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results.
Field Duplicate (See Note #7 for additional information.)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are ≥5× RL. Use limit of ± RL (±2× RL for solids) when at least one sample value is <5× RL. (Use one-half the RL as a numerical value for any "not-detected" results in the RPD calculations)..	If the criteria are not met, qualify positive results for the non-compliant compound in the original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Percent Solids	Solid samples with less than 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.

Table for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Quality Control Item	Usability Criteria	Action
Compound Quantitation (See Note #8 for additional information.)	Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution.	If a target compound result exceeds the instrument calibration range, qualify positive results as estimated ("J"). Use professional judgement when evaluating sample reanalyses and dilutions. If criteria (see field duplicate usability) between the original sample results and the reanalysis sample results are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a target compound result is <RL but ≥MDL, qualify positive results as estimated ("J").
System Performance (See Note #9 for additional information.)	Professional judgement should be used when assessing the degradation of system performance during analyses.	Use professional judgement to qualify the data if it is determined that system performance degraded during sample analyses.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitations of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

1. $$\%\ \text{breakdown for 4,4′ - DDT} = \frac{\text{Total DDT degradation peak area (DDE+ DDD)}}{\text{peak areas (DDT + DDE +DDD)}} \times 100$$

$$\%\ \text{breakdown for Endrin} = \frac{\text{Total Endrin degradation peak area (Endrin aldehyde + Endrin ketone)}}{\text{peak areas (Endrin + Endrin aldehyde + Endrin ketone)}} \times 100$$

Qualification Due to High 4,4′-DDT (or Endrin) Breakdown				
Column # / Met criteria?	If 4,4′-DDT (or Endrin):	And if 4,4′-DDD and/or 4,4′-DDE (or Endrin ketone and/or Endrin aldehyde):	Then flag 4,4′-DDT (or Endrin):	And flag **positives** for 4,4′-DDD and/or 4,4′-DDE (or Endrin ketone and/or Endrin aldehyde):
(1)/no (2)/yes	+ +	any +'s +'s or ND's	"J" column (1) OK column (2)	"JN" column (1) "N" column (2)
(1)/no (2)/yes	ND + > RL^+	any +'s +'s or ND's	ND "R"	"JN" column (1) "N" column (2)
(1)/no (2)/yes	ND + < RL^+	any +'s +'s or ND's	ND OK	"JN" column (1) "N" column (2)
(1)/no (2)/yes	+ +	all ND's +'s or ND's	*	NA
(1)/no (2)/yes	ND + or ND	all ND's +'s or ND's	ND OK	NA
(1)/no (2)/yes	+ or ND ND	+'s or ND's +'s or ND's	ND OK	OK
(1)/no (2)/no	ND + or ND	any +'s any +'s	ND "R"	"JN"
(1)/no (2)/no	+ +	any +'s any +'s	"J"	"JN"
(1)/no (2)/no	+ ND	all ND's any +'s	ND "R"**	NA
(1)/no (2)/no	+ +	all ND's all ND's	*	NA
(1)/no (2)/no	+ or ND ND	+'s or ND's all ND's	ND OK	NA

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Where:

+ A peak was observed in the RT window for this pesticide on the corresponding GC column indicating a tentative identification for this pesticide. The positive result may quantitate to be below, at, or above the RL.

ND Not Detected regardless of the RL (flat baseline was observed in the area of the chromatogram where this compound would elute if it were truly present in the sample).

RL Reporting limit (RL) – will be defined on a project basis. If the project-required RL is greater than the low initial calibration standard concentration, the Project Manager should be consulted about application of qualifications related to the RL. Positives from a non-quantitative (conformational) GC column analysis should be considered above the RL for evaluation purposes, whether or not the result was quantitated above the RL on this non-quantitative column.

NA Not Applicable. Pesticide was not detected and only positive results are impacted.

* Although high breakdown was indicated by the associated standard on at least one column used for analysis, this positive result for 4,4′-DDT (or Endrin) has <u>not</u> been qualified because the breakdown components were not detected in the sample analysis on the noncompliant column(s). It is questionable, however, whether the peak(s) used for identification on the noncompliant column(s) truly represents 4,4′-DDT (or Endrin)

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

because high breakdown was indicated by the associated standard. It is highly unusual not to detect the breakdown components in the presence of 4,4′-DDT (or Endrin).

** This "not-detected" result for 4,4′-DDT (or Endrin) has been qualified as unusable ("R") because the breakdown components were observed in the sample analysis on this column on which high breakdown was indicated by the associated standard. However, it should be noted that the breakdown components were <u>not</u> detected in the sample analysis on the other column on which high breakdown was also indicated by the associated standard. It is questionable whether the peak used for identification on this other column truly represents 4,4′-DDT (or Endrin) because high breakdown was indicated by the associated standard on this other column. It is highly unusual not to detect the breakdown components in the presence of 4,4′-DDT (or Endrin).

2. If the initial calibration curve %RSD>50%, the linearity of the first three initial calibration standards should be evaluated. If the first three initial calibration standards for the compound are linear (*i.e.*, r > 0.99), do not qualify "not-detected" results. If the first three initial calibration standards for the compound are not linear, qualify "not-detected" results as estimated ("UJ").

Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive, the samples should be evaluated for false positives. If the concentration intercept is negative, the sample should be evaluated for false negatives.

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Positive results should only be qualified if the results were reported from the out-of-criteria column. If samples are analyzed on two columns and an out-of-criteria initial calibration is reported on either column, the sample should be evaluated for tentative positive results. If a tentative positive result is observed on the compliant column, qualify "not-detected" results (laboratory should only report positive results that have been confirmed on a second column) as estimated ("UJ"). If an out-of-criteria initial calibration is reported on both columns, qualify "not-detected" results as estimated ("UJ") whether or not tentative positive results were observed.

3. If instrument instability (*i.e.*, several continuing calibration standards with compounds exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential continuing calibration standards, "not-detected" results may be qualified as estimated ("UJ") due to instrument sensitivity of a continuing calibration standard response that is greater than the initial calibration standard response (increase in instrument sensitivity).

   If the continuing calibration standard is %D>15% in the direction of increased instrument sensitivity and it is determined that "not-detected" results should not be qualified, the data reviewer should note this within the QAR support documentation.

   Positive results should only be qualified if the results were reported for the out-of-criteria column. If samples are analyzed on two columns and an out-of-criteria CCV is reported on either column, the samples should be evaluated for tentative positive results. If a tentative positive result is observed on the compliant column, qualify "not-detected"

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

results (laboratory should only report positive results that have been confirmed on a second column) as estimated ("UJ"). If an out of criteria CCV is reported on both columns, qualify "not-detected" results as estimated ("UJ") whether or not tentative positive results were observed.

4. Use professional judgement when evaluating sample chromatograms. If the chromatograms reveal peaks corresponding to target compounds of interest using expanded RT windows, reported positive sample results for the compound outside of the RT window are replaced with the RL and are qualified as "not-detected" ("U"). If the chromatograms reveal peaks that interfere with potential detection of a target compound, reported positive results for that compound are qualified as unusable ("R").

5. The frequency of field/equipment/rinse blanks is determined during the sampling event. The results of a field/equipment/rinse blank should be applied to all samples collected on the same day, unless only one blank was collected for a several-day sampling event. In instances when more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant.

   If a sample result qualified "U*" is <RL and the laboratory did not report the RL on the data tables or Form I, the positive result (*e.g.*, 8 µg/L) should be replaced with the RL (*e.g.,* 10 µg/L).

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

Instrument blank contamination should be applied to samples bracketing the contaminated instrument blank. If a positive result for a target compound is detected on the first column and not on the second column, the positive results should be qualified as tentative in all associated samples ("N"). Use professional judgement to determine if the result should be qualified as "not-detected" ("U*").

6. The surrogate recovery limits do not apply to samples analyzed at greater than five-fold dilutions. Qualification of the data is not necessary if the surrogate is diluted beyond detection. Generally, a greater than five-fold dilution will affect the ability to even detect the surrogate. If a sample was analyzed at a five-fold dilution or less and either surrogate was not detected in the sample, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Write a comment in the QAR addressing the issue that sample-specific method performance based on surrogate recoveries could not be evaluated due to the dilution required for sample analysis.

7. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. Laboratory duplicate results and field duplicate results apply only to the original sample and the laboratory/field duplicate. Soil duplicate results will have greater variance than aqueous duplicate results.

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

8. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

   If the laboratory provides the dual-column results for samples and both columns are quantitative, evaluate the %D between the results reported for both columns. If the sample results are ≥5× RL and the %D is >40% but ≤90%, qualify positive results as estimated ("J"). If the sample results are ≥5× RL and the %D is >90%, qualify positive results as unusable ("R"). If sample results are <5× RL and the difference between columns is >±2×RL, qualify results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

9. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

   - high background levels or shifts in absolute RTs of internal standards
   - excessive baseline rise at elevated temperature
   - extraneous peaks
   - loss of resolution
   - peak tailing or peak splitting that may result in inaccurate quantitation

Notes for the Validation of Organochlorine Pesticide Data Generated by SW-846 Method 8081A

10. The RL will be defined on a project-specific basis. If the project-required RL is less than the low calibration standard concentration, the Project Manager should be consulted for instructions about application of qualification related to the RL.

APPENDIX 35

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate herbicide data generated by SW-846 Method 8151A for the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to SW-846 Method 8151A and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the herbicide data provided by the analytical laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory National Functional Guidelines for Organic Data Review" (10/99; National Functional Guidelines). It should be noted that National Functional Guidelines apply strictly to data generated by Contract Laboratory Program (CLP) protocol and it is not directly applicable to validation of data generated by SW-846 Method 8151A; this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes, or a data summary spreadsheet of the qualified analytical results will be included in the support documentation of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCEL forms:

- Organic field duplicate comparison Rev 1-01.xls
- Organic field quadruplicate comparison Rev 1-01.xls
- Organic field triplicate comparison Rev 1-01.xls

Chemistry Applications:

- FIT
- Methods Database
- Target version 4.1 data processing software

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/99).

- SW-846 Method 8151A and related preparation and cleanup methods.

- Region I, EPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses.

- Region III, Modifications to National Functional Guidelines for Organic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the herbicide data based on an evaluation of information presented in the data package deliverables. Compliance to SW-846 Method 8151A and/or other reference documents (*e.g.,* analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratory(ies) to request the correction of deficiencies prior to submittal of the QAR (if feasible and sanctioned by General Electric

Company). At a minimum, corrections required to allow for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the comments subsection of the QAR and will be included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the herbicide data based on an evaluation of the information presented in the data package deliverables. The findings of the herbicide data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data; these qualifications will be presented in the Organic Data Qualifier subsection of the Organic Data Evaluation Section of the QAR. Each qualification will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) in General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination, unusable results (R/UR), tentative identifications of target compounds (N), estimated results (J/UJ), field duplicate comparison, and a general qualifier for all results reported below the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the herbicide data and the resultant qualifications will be as stated on the attached Table for the Validation of

Herbicide Data Generated by SW-846 method 8151A. It should be noted that the Project Manager should be consulted when the use of "professional judgement" is indicated on the attached table.

Table for the Validation of Herbicide Data Generated by SW-846 Method 8151A

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	4±2°C	If temperature is >10°C, but ≤20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as "UJ". If temperature is >20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Note time of collection relative to receipt at laboratory; use professional judgement if < 8 hours has elapsed from collect to receipt to determine if the qualification for elevated temperature applies.
Technical Holding Time	Aqueous samples should be extracted within 7 days of sample collection. Solid samples should be extracted within 14 days of sample collection. All matrices should be analyzed within 40 days after extraction.	If holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results ("UJ"). If holding time is grossly exceeded (*i.e.,* >twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Herbicide Data Generated by SW-846 Method 8151A

Quality Control Item	Usability Criteria	Action
Initial Calibration (See Note #1 for additional information.)	%RSD should be <20% or a calibration curve should be generated. If a curve is generated, r (linear) or coefficient of determination (COD; quadratic) should be ≥0.99.	If any target compound result is associated with a low concentration initial standard that is not visible on the chromatogram, qualify "not-detected" results for that compound as estimated ("UJ"). If the standards indicate a severe lack of sensitivity (*e.g.*, the higher calibration standards are barely visible) the reviewer may qualify the "not-detected" results for that compound as unusable ("UR"); professional judgement should be used to determine the magnitude of the bias. Compounds with 20%<%RSD≤50%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. Compounds with 50%<%RSD≤90%, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. (See Note #1) Compounds with %RSD >90%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Use professional judgement when evaluating correlation coefficients (r) and coefficients of determination (COD). If r (linear) or COD (quadratic) <0.99 but ≥0.85, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If r or COD is <0.85, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the initial calibration standards and the samples associated were not performed similarly (*e.g.*, the initial calibration standards were analyzed under different chromatographic conditions), qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

Table for the Validation of Herbicide Data Generated by SW-846 Method 8151A

Quality Control Item	Usability Criteria	Action
Continuing Calibration Verification (CCV) (See Note #2 for additional information.)	%drift or % difference (%D) should be ≤15%.	Qualification is for all samples on both sides of the out of criteria CCV. If target compounds have 15%<%D≤90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If target compounds have %D>15% with the response indicating a sensitivity increase, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. (See Note #2) If target compounds have %D>90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If any target compound is not visible in the chromatogram qualify "not-detected" results as unusable ("UR").
Internal Standards (If used)	Area counts of the internal standard peaks should be 50-200% of the area of the target analytes in the mid-point calibration analysis. RT for any internal standard changes should only be ±30 seconds from the last calibration check standard.	If an area count is outside of the criteria (50-200%), qualify positive results for compounds quantitated using that internal standard as estimated ("J") and qualify "not-detected" results for compounds quantitated using that internal standard as estimated ("UJ"). If extremely low area counts (<25%) are reported, qualify positive results for compounds quantitated using the extremely low internal standard as estimated ("J") and qualify "not-detected" results for compounds using that internal standard as unusable ("UR"). If an internal standard RT varies by more than 30 seconds and no peaks are observed in the sample chromatogram, qualification of data is not necessary. Use professional judgement if peaks are observed in the sample chromatogram.

Table for the Validation of Herbicide Data Generated by SW-846 Method 8151A

Quality Control Item	Usability Criteria	Action
Retention Time Windows (See Note #3 for additional information.)	All target compound retention times (RTs) should be within the established RT windows. RT windows should be estimated or defined by the laboratory or 3× the standard deviation of three non-sequential standards over a 72-hour period	If the CCV RT windows are not within the specific RT windows, evaluate sample chromatograms for false positives and false negatives. If a constant drift in RT is observed in the bracketing CCV, apply the direction of the RT drift to the sample chromatograms.
Blanks (See Notes #4 and #9 for additional information.)	Summarize all results greater than the method detection limit (MDL) in the blanks. The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a target compound is detected in a blank but not in the associated sample(s), no action is required. If a sample result is ≤5× the blank result, qualify the positive result as "not-detected" ("U*"). If the positive result qualified "U*" is <RL, the RL should be used as reported. If the positive result qualified "U*" is ≥RL, the value of the positive result should be used as the revised RL. If a sample result is >5× the blank result, qualification is not required. If gross contamination exists (*i.e.*, saturated peaks on the GC), qualify the positive results as unusable ("R") due to interference.
Surrogates (See Note #5 for additional information.)	Use laboratory acceptance limits. Use 20-150% as default limits if the laboratory limits are unreasonable.	If the recoveries of one or more surrogate are > upper limit, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the recoveries of one or more surrogate are < lower limit but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recoveries of one or more surrogate are <10%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Herbicide Data Generated by SW-846 Method 8151A

Quality Control Item	Usability Criteria	Action
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	For accuracy, use recovery limits of 50-135%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPD calculated on %Rs) that are outside of criteria if the original concentration of a compound is >4× spiking level for that compound. RPDs calculated using MS/MSD results can be used to evaluate precision. If the recovery > 135%, qualify the positive result in the native sample as estimated ("J") and do not qualify "not-detected" results. If the recovery < 50% but ≥10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native samples estimated ("UJ"). If the recovery < 10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive result in the native sample as estimated ("J") and do not qualify "not-detected" results. If the precision criteria (see field duplicate usability criteria) are not met for non-spiked compounds, qualify positive results in the native sample as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a field duplicate of the native sample was collected and analyzed, the field duplicate sample should also be qualified if the MS/MSD recoveries or RPD are outside of the criteria (as stated above for the native sample).

Table for the Validation of Herbicide Data Generated by SW-846 Method 8151A

Quality Control Item	Usability Criteria	Action
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	For accuracy, use recovery limits of 50%-135%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	If the recovery > 135%, qualify positive results for that compound in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery < 50% but ≥ 10%, qualify positive results for that compound in all associated samples as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recovery <10%, qualify positive results for that compound in all associated samples as estimated and qualify "not-detected" results as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive results for that compound in all associated samples as estimated ("J") and do not qualify "not-detected" results.
Field/Laboratory Duplicate (See Note #6 for additional information.)	Use a default limit of 20% RPD (%RSD for triplicate or quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate or quadruplicate analyses) for solid samples for sample results ≥5× RL. Use a default limit of ± RL for aqueous samples and ±2× RL for solid samples when at least one sample value is <5× RL	If the criteria are not met, qualify positive results for out of criteria compounds in the original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Percent Solids	Solid samples with <50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.

Table for the Validation of Herbicide Data Generated by SW-846 Method 8151A

Quality Control Item	Usability Criteria	Action
Compound Quantitation (See Note #7 for additional information.)	Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution.	If a target compound result exceeds the instrument calibration range, qualify positive results as estimated ("J"). If a target compound result is <RL but ≥MDL, qualify positive results as estimated ("J"). Use professional judgement to determine whether sample reanalyses and dilutions should be compared to the original analysis. If the precision criteria (see field duplicate usability) between the original sample results and the reanalysis sample result is not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
System Performance (See Note #8 for additional information.)	Professional judgement should be used when assessing the degradation of the system performance during analyses.	Use professional judgement to qualify the data if it is determined that the system performance degraded during sample analyses.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitations of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

Notes for the Validation of Herbicide Data Generated by SW-8151A

1. If the initial calibration %RSD is >50%, the linearity of the first three initial calibration standards for the compound should be evaluated. If the first three initial calibration standards for the compound are linear (*i.e.*, r ≥ 0.99), do not qualify "not-detected" results. If the first thee initial calibration standards for the compound are not linear, qualify "not-detected" results as estimated ("UJ").

   Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive, the samples should be evaluated for false positives. If the concentration intercept is negative, the samples should be evaluated for false negatives.

2. If instrument instability (*i.e.*, several CCVs with compounds exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential CCVs, "not-detected" results may be qualified as estimated ("UJ") due to instrument sensitivity of a continuing calibration standard response that is greater than the initial calibration standard response (increase in instrument sensitivity).

   If the continuing calibration standard is %D>15% in the direction of increased instrument sensitivity and it is determined that "not-detected" results should not be qualified, the data reviewer should note this in the QAR support documentation.

   Positive results should only be qualified if the results were reported from the out of criteria column. If samples are analyzed on two columns and an out of criteria CCV is reported on either column, then the samples should be evaluated for tentative positive

Notes for the Validation of Herbicide Data Generated by SW-8151A

results. If a tentative positive result is observed on the compliant column, qualify "not-detected" results (laboratory should only report positive results that have been confirmed on a second column) as estimated ("UJ"). If an out of criteria CCV is reported on both columns, qualify "not-detected" results as estimated ("UJ") whether or not tentative positive results were observed.

3. Use professional judgement when evaluating sample chromatograms. If the chromatograms reveal peaks corresponding to target compounds of interest using expanded RT windows, reported positive sample results for the compound outside the RT window are replaced with the RL and qualified as "not-detected" ("U"). If chromatograms reveal peaks that interfere with potential detection of a target compound, reported positive results for the compound are qualified as unusable ("R").

4. When samples that are extracted together are analyzed on separate instruments or on separate analytical shifts, the method blank associated with those samples should be analyzed on at least one of those instruments. A solvent blank should be analyzed on all other instruments on which the set of samples was analyzed to demonstrate that the instrument is not contributing contaminants to the samples.

   The frequency of equipment/rinse blanks is determined during the sampling event. The results of equipment/rinse blanks should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day; however, if only one was collected for a several-day sampling event. In instances when more than one blank is

Notes for the Validation of Herbicide Data Generated by SW-8151A

associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration for a contaminant.

If a sample result qualified "U*" is less than the RL and the laboratory did not report the RL on the data tables or Form I's, then the positive result (*e.g.*, 8 µg/L) should be replaced with the RL (*e.g.*, 10 µg/L).

5. The surrogate recovery limits do not apply to samples analyzed at dilutions greater than five-fold. Qualification of the data is not necessary if the surrogate is diluted beyond detection. Generally, greater than a five-fold dilution will affect the ability to even detect the surrogate. If a sample was analyzed at five-fold dilution or less and the surrogate was not detected in the sample, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

6. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. Field duplicate sample results should only be applied to the original sample and its field duplicate. Laboratory duplicates should only be applied to the original sample and its laboratory duplicate. It is also expected that soil duplicate results will have a greater variance than aqueous duplicate results.

Notes for the Validation of Herbicide Data Generated by SW-8151A

7. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

   If the laboratory provides dual column results for samples and both columns are quantitative then the %D between the results reported for both columns needs to be evaluated. If sample results are ≥5× RL and the %D is >40% but ≤90%, qualify positive results as estimated ("J"). If sample results are >5× RL and the %Ds is >90%, qualify positive results as unusable ("R"). If sample results are <5× RL and the differences between columns is >±2×RL, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

8. Poor chromatographic performance affects both qualitative (lower dilution analyses) and quantitative results. Indications of substandard performance include:

   - high background levels or shifts in absolute retention times of internal standards
   - excessive baseline rise at elevated temperature
   - extraneous peaks
   - loss of resolution
   - peak tailing or peak splitting that may result in inaccurate quantitation

9. The RL will be defined on a project-specific basis. If the project-required RL is less than the low initial calibration standard concentration, the Project Manager should be consulted for instructions on how to apply qualification related to the RL.

APPENDIX 36

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate PCB data generated by SW-846 Method 8082 for the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to SW-846 Method 8082 and/or other reference documents (*e.g.*, analytical SOPs) as applicable to the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the PCB data provided by the analytical laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory National Functional Guidelines for Organic Data Review" (10/99; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by the Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by SW-846 Method 8082; therefore, this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes or a data summary spreadsheet of the qualified analytical results, will be included in the support documentation of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELforms:

- Organic field duplicate comparisons Rev1-01.xls
- Organic field quadruplicate comparison Rev1-01.xls
- Organic field triplicate comparison Rev1-01.xls
- Aroclor.xls

Chemistry Applications:

- FIT
- Methods Database
- Target version 4.1 data processing software

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/99).

- SW-846 Method 8082.

- Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, Standard Operating Procedure for the Validation of Organic Data Acquired Using SW-846 Method 8082 (Rev 2. 12/96).

- Region III, Modifications to National Functional Guidelines for Organic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the PCB data based on an evaluation of information presented in the data package deliverables. Compliance to SW-846 Method 8082 and/or other reference documents (*e.g.,* analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of deficiencies prior to submittal of the QAR (if feasible and sanctioned by to General Electric Company). At a minimum, corrections required to allow for a full evaluation of

the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the comments subdivision of the QAR and included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the PCB data based on an evaluation of the information presented in the data package deliverables. The findings of the PCB data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data; these qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) to General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination (U*), unusable results (R/UR), estimated results (J/UJ), tentative identifications of target compound results (N), field duplicate comparison, and a general qualifier for all results reported below the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the PCB data and the resultant qualifications will be as stipulated on the attached Table for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082. It should be noted that the project

manager should be consulted when "professional judgement" use is indicated on the attached table.

Table for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	4±2°C	If temperature is >10°C but ≤20°C, qualify positive results as estimated ("J") and qualify "not-detected results as estimated ("UJ"). If temperature is > 20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Note time of collection relative to receipt at laboratory. Professional judgement should be used if < 8 hours has elapsed from collection to receipt at the laboratory to determine if qualification due to elevated temperature applies.
Technical Holding Time	Aqueous samples should be extracted within 7 days of sample collection. Solid/soil samples should be extracted within 14 days of sample collection. All matrices should be analyzed within 40 days after extraction.	If a holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a holding time is grossly exceeded (*i.e.*, > twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Initial Calibration (See Note #1 for additional information.)	%RSD should be ≤20% and a calibration curve should be generated. For the calibration curve, r (linear) or coefficient of determination (COD; quadratic) must be ≥0.99.	If target Aroclors have 20%< %RSD ≤50%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If target Aroclors have 50%< %RSD ≤90%, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. If target Aroclors have %RSD > 90%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Use professional judgement when evaluating correlation coefficients (r) and coefficients of determination (COD). If r (linear) or COD (quadratic) is <0.99 but ≥0.85, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If r or COD is <0.85, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

Quality Control Item	Usability Criteria	Action
Continuing Calibration Verification (CCV) (See Note #2 for additional information.)	%drift or %difference should be ≤15%.	Qualification is for all samples on both sides of the out-of-criteria calibration standards. If target Aroclors have 15%<%D≤90% with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If target Aroclors have %D>15% with the response indicating a sensitivity increase, qualify positive results as estimated ("J") and use professional judgement to qualify "not-detected" results. If target Aroclors have %D>90% with the response indicating a sensitivity decrease qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Retention Time Windows (See Note #3 for additional information.)	All target Aroclor peak retention times (RTs) should be within the established RT windows. RT windows should be estimated or defined by the laboratory or 3× the standard deviation of three non-sequential standards over a 72-hour period.	If the CCV RT windows are not within the specified RT windows, evaluate sample chromatograms for false positives and false negatives. If a constant drift in RT is observed in the bracketing CCV, the direction of the RT drift should be applied to the sample chromatograms.
Blanks (See Note #4 and Note #9 for additional information.)	Summarize all results greater than the method detection limit (MDL) present in the blanks. The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a target Aroclor is found in the blank but not in the associated sample(s), no action is required. If a sample result is ≤5× the blank result, qualify the positive result as "not detected" ("U*"). If the positive result qualified "U*" is <RL, the RL should be used as reported. If the positive result qualified "U*" is ≥RL, the value of the positive result should be used as the revised RL. If a sample result is >5× blank result, qualification is not required. If gross contamination exists (*i.e.*, saturated peaks on the GC), qualify the positive results as unusable ("R") due to interference.

Table for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

Quality Control Item	Usability Criteria	Action
Surrogates (See Note #5 for additional information.)	Use 60-140% as limits.	If the recoveries of one or more surrogates are > upper limit, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the recoveries of one or more surrogates are < lower limit but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recoveries of one or more surrogates are <10%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Matrix Spike/Matrix Spike Duplicate (MS/MSD) (IF REQUESTED)	For accuracy, use recovery limits of 60-140%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if the original concentration of an Aroclor is >4× the spiking level for that compound. RPDs calculated using MS/MSD results can be used to evaluate precision. If the recovery is >140%, qualify the positive result in the native sample as estimated ("J") and do not qualify the "not-detected" result. If the recovery is <60% but ≥10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as estimated ("UJ"). If the recovery is <10%, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native sample as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive result in the native sample as estimated ("J") and do not qualify the "not-detected" result. If the precision criteria (see field duplicate usability criteria) for non-spiked compounds are not met, qualify the positive result in the native sample as estimated ("J") and qualify the "not-detected" result in the native samples as estimated ("UJ"). If a field duplicate of the native sample was collected and analyzed, the field duplicate sample should also be qualified if an MS/MSD recovery or RPD is outside of criteria (as stated above for the native sample).

Table for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

Quality Control Item	Usability Criteria	Action
Laboratory Control Samples (LCS)	For accuracy, use recovery limits of 60-140%.	If the recovery is >140%, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery is <60% but ≥10%, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as estimated ("UJ"). If the recovery is <10%, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as unusable ("UR").
Field/Laboratory Duplicate (See Note #6 and Note #9 for additional information)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are ≥5× RL. Use limit of ± RL (±2× RL for solids) when at least one sample value is <5× RL. (Use one-half the RL as a numerical value for any "not-detected" results in the RPD calculations).	If the criteria are not met, qualify positive results for the out-of-criteria compounds in the original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Percent Solids	Solid samples with less than 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.

Table for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

Quality Control Item	Usability Criteria	Action
Compound Quantitation and Qualititative Identification (See Notes #3, #7, and #8 for additional information.)	Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution. All sample chromatograms must be evaluated to determine whether the laboratory correctly identified the correct Aroclor based upon pattern recognition, peak retention times, and qualititative matching with the associated calibration standards.	If a target Aroclor result exceeds the instrument calibration range, qualify positive result as estimated ("J"). Use professional judgement to determine whether sample reanalyses and dilutions should be compared to the original analyses. If criteria (see field duplicate usability) between the original sample results and the reanalysis sample results are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a target Aroclor is <RL but ≥MDL, qualify positive results as estimated ("J"). Use professional judgement to determine whether qualititative identifications are accurate and whether data qualification is necessary.
System Performance (See Note #8 for additional information.)	Professional judgement should be used when assessing the degradation of system performance during analyses.	Use professional judgement to qualify the data if it is determined that system performance degraded during sample analyses.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitations of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

Notes for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

1. If the initial calibration curve %RSD>50%, the linearity of the first three initial calibration standards should be evaluated. If the first three initial calibration standards for the compound are linear (*i.e.*, r ≥0.99), do not qualify "not-detected" results. If the first three initial calibration standards for the compound are not linear, qualify "not-detected" results as estimated ("UJ").

   Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive, the samples should be evaluated for false positives. If the concentration intercept is negative, the sample should be evaluated for false negatives.

   The initial calibration is performed using five-point initial calibration curves for Aroclor-1221, Aroclor-1242, and Aroclor-1254 because these Aroclors are the target compounds for the project. Single-point calibrations for each of the other Aroclors should be analyzed at or just above the quantitation limits for pattern recognition. Curves (linear not through the origin or a quadratic) should be generated and relative standard deviations (%RSDs) must be ≤20%. If positive results are detected, other than the three Aroclors in the multi-peak initial calibration curves, in the samples, then a five-point initial calibration curve should be analyzed for the Aroclor in question and the extracts must be re-injected. Surrogates will be added to the Aroclor-1254 initial calibration curve and all surrogates in the samples/blanks/QC samples/CCVs will be quantitated based on the Aroclor-1254 initial calibration.

Notes for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

2. If instrument instability (*i.e.*, several continuing calibration standards with compounds exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential continuing calibration standards, "not-detected" results may be qualified as estimated ("UJ") due to instrument sensitivity of a continuing calibration standard response that is greater than the initial calibration standard response (increase in instrument sensitivity).

   If the continuing calibration standard is %D>15% in the direction of increased instrument sensitivity and it is determined that "not-detected" results should not be qualified, the data reviewer should note this within the QAR support documentation.

   The continuing calibration verification standard analysis utilizes only Aroclor-1221, Aroclor-1242, and Aroclor-1254; therefore, if the %D>15% for these three Aroclors, the impact on the other Aroclors (-1016, -1232, -1248, and -1260) should be evaluated. The impact depends upon the retention times of the other Aroclors. The retention times should be evaluated to determine which Aroclor (-1221, -1242, or -1254) will affect the other Aroclors. Usually, Aroclors-1016, Aroclor-1221, Aroclor-1232, Aroclor-1242, and Aroclor-1248 fall within similar retention times and Aroclor-1248, Aroclor-1254, and Aroclor-1260 fall within similar retention times; therefore, if %D>15% for Aroclor-1242 or Aroclor-1221, then –1016, -1232, and -1248 should be qualified (as stated above), and if %D>15% for Aroclor-1254, then Aroclor-1248 and Aroclor-1260 should be qualified (as stated above).

Notes for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

3. Use professional judgement when evaluating sample chromatograms. The Aroclor patterns should be evaluated when an RT shift is observed. If the Aroclor pattern is present, qualification is not necessary. If the chromatograms reveal peaks corresponding to target compounds of interest using expanded RT windows and the surrogate compounds do not display a similar shift in RT, reported positive sample results for the compound outside of the RT window are replaced with the RL and qualified as "not-detected" ("U").

   If the chromatograms reveal peaks that interfere with potential detection of a target compound, qualify reported positive results for the compound as unusable ("R").

4. The frequency of equipment/rinse blanks is determined during the sampling event. The results of a equipment/rinse blank should be applied to all samples collected in the same day, unless only one blank was collected for a several-day sampling event. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant.

   If a sample result qualified "U*" is <RL and the laboratory did not report the RL on the data tables or Form I, the positive result (*e.g.*, 8 µg/L) should be replaced with the RL (*e.g.*, 10 µg/L).

   Instrument blank contamination should be applied to samples bracketing the contaminated instrument blank.

Notes for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

5. The surrogate recovery limits do not apply to samples analyzed at greater than five-fold dilutions. Qualification of the data is not necessary if the surrogate is diluted beyond detection. Generally, a greater than five-fold dilution will affect the ability to even detect the surrogate. If a sample was analyzed at a five-fold dilution or less and either surrogate was not detected in the sample, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Write a comment in the QAR addressing the issue that sample-specific method performance based on surrogate recoveries could not be evaluated due to the dilution required for sample analysis.

6. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. Laboratory duplicate results and field duplicate results apply only to the original sample and the laboratory/field duplicate. Soil duplicate results are expected to have greater variance than aqueous duplicate results.

7. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

8. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

   - High background levels or shifts in absolute RTs of internal standards

Notes for the Validation of PCB (Aroclor) Data Generated by SW-846 Method 8082

- Excessive baseline rise at elevated temperature
- Extraneous peaks
- Loss of resolution
- Peak tailing or peak splitting that may result in inaccurate quantitation

9. The RL will be defined on a project-specific basis. If the project-required RL is less than the low calibration standard concentration, the Project Manager should be consulted for instructions about application of qualification related to the RL.

APPENDIX 37

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate PCB data generated by US EPA Method 680 for the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to US EPA Method 680 and/or other reference documents (*e.g.*, analytical SOPs) as applicable to the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the PCB data provided by the analytical laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory National Functional Guidelines for Organic Data Review" (10/99; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by the Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by US EPA Method 680; therefore, this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes or a data summary spreadsheet of the qualified analytical results, will be included in the support documentation of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELforms:

- Organic field duplicate comparisons Rev1-01.xls
- Organic field quadruplicate comparison Rev1-01.xls
- Organic field triplicate comparison Rev1-01.xls

Chemistry Applications:

- FIT
- Methods Database
- Target version 4.1 data processing software

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/99).

- US EPA Method 680 as presented in SOP GEHR680.

- Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, Standard Operating Procedure for the Validation of Organic Data Acquired Using SW-846 Method 8082 (Rev 2. 12/96).

- Region III, Modifications to National Functional Guidelines for Organic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the PCB data based on an evaluation of information presented in the data package deliverables. Compliance to US EPA Method 680 and/or other reference documents (*e.g.,* analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/ comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of deficiencies prior to submittal of the QAR (if feasible and sanctioned by to General Electric Company). At a minimum, corrections required to allow for a full evaluation of

the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the comments subdivision of the QAR and included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the PCB data based on an evaluation of the information presented in the data package deliverables. The findings of the PCB data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data; these qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) to General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination (U*), unusable results (R/UR), estimated results (J/UJ), tentative identifications of target compound results (N), field duplicate comparison, and a general qualifier for all results reported below the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the PCB data and the resultant qualifications will be as stipulated on the attached Table for the Validation of PCB (Aroclor) Data Generated by US EPA Method 680 (SOP GEHR680). It should be noted

that the project manager should be consulted when "professional judgement" use is indicated on the attached table.

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	4±2°C	If temperature is >10°C but ≤20°C, qualify positive results as estimated ("J") and qualify "not-detected results as estimated ("UJ"). If temperature is > 20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Note time of collection relative to receipt at laboratory. Professional judgement should be used if < 8 hours has elapsed from collection to receipt at the laboratory to determine if qualification due to elevated temperature applies.
Technical Holding Time	Aqueous samples should be extracted within 7 days of sample collection. Solid/soil samples should be extracted within 14 days of sample collection. All matrices should be analyzed within 40 days after extraction.	If a holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If a holding time is grossly exceeded (*i.e.*, > twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
GC/MS Tuning (DFTPP)	Ion abundances should meet the method acceptance criteria. (See Note #1 for criteria.)	If mass calibration was not performed, qualify all associated data as unusable ("R"). If mass assignment is in error, qualify all associated data as unusable ("R"). Use professional judgment if abundance criteria are not met. Use professional judgment if samples are analyzed more than 12 hours after a compliant tune and there is no evidence of a compliant tune following the samples.

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Initial Calibration (See Note #2 for additional information.)	Nine selected PCB congeners are used as calibration standards to represent each homolog group, the mono- through octachlorobiphenyls and decachlorobiphenyl. Decachlorobiphenyl is used as the calibration congener for both nona- and decachlorobiphenyl homolog groups. Five response factors (RFs) for each PCB calibration congener and surrogate must be calculated relative to chrysene-d_{12}. If interference or problems exist with chrysene-d_{12} then RFs will be calculated using phenanthrene-d_{10}. Each %RSD should be ≤20%.	If a PCB congener has 20%< %RSD ≤50%, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and do not qualify "not-detected" results for the associated homolog group(s) and total PCBs. If a PCB congener has 50%< %RSD ≤90%, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and use professional judgement to qualify "not-detected" results for the associated homolog group(s) and total PCBs. If a PCB congener has %RSD > 90%, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and qualify "not-detected" results for the associated homolog group(s) and total PCBs as unusable ("UR").
Continuing Calibration Verification (CCV) (See Note #3 for additional information.)	A CCV is required at the beginning and end of each 12-h period during which analyses are performed. The % difference (%D) for each PCB calibration congener and surrogate in each CCV should be ≤20%.	Qualification is for all samples on both sides of the out-of-criteria calibration standards. If a PCB congener has 20%<%D≤90% with the response indicating a sensitivity decrease, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and qualify "not-detected" results for the associated homolog group(s) and total PCBs as estimated ("UJ"). If a PCB congener has %D>20% with the response indicating a sensitivity increase, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and use professional judgement to qualify "not-detected" results for the associated homolog group(s) and total PCBs. If a PCB congener has %D>90% with the response indicating a sensitivity decrease qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and qualify "not-detected" results for the associated homolog group(s) and total PCBs as unusable ("UR").

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
SIM PCB Data Performance Criteria for Calibration Standards	GC separation -- Baseline separation of PCB congener #87 (Cl_5) from congeners #154 (Cl_6) and #77 (Cl_4), which may coelute.	If baseline separation is not observed and the unresolved congeners are observed in an associated sample, qualify positive results for the associated homolog groups and total PCBs as estimated ("J").
	MS sensitivity -- Signal/noise ratio of ≥ 5 for m/z 499 of PCB congener #209, Cl_{10} -PCB, and for m/z 241 of chrysene-d_{12}.	If the S/N ratio was <5, use professional judgment to determine potential qualitative impacts.
	MS calibration -- Abundance of ≥ 70% and ≤ 95% of m/z 500 relative to m/z 498 for congener #209, Cl_{10} -PCB.	If the relative ion abundance ratio was not with the stated range, use professional judgment to determine the accuracy of qualitative identifications (both positive and "not-detected" results), focusing on chlorine cluster ions. Carefully evaluate sample ion ratios.
Internal Standards	The area measured for m/z 240 for chrysene-d_{12} nor that for m/z 188 for phenanthrene-d_{10} should not have changed by more than 30% from the area measured in the most recent previous analysis of a CCV standard or decreased by more than 50% from the mean area measured during initial calibration. Retention time (RT) of the internal standard should not vary more than ±10 seconds from the RT of the internal standards observed in associated CCV standard.	If a sample area count is outside of the criteria (70-130% of associated CCV or ≥50% of the associated ICV), qualify positive results for the homolog groups quantitated using that internal standard and total PCBs as estimated ("J") and qualify "not-detected" results for the homolog groups quantitated using that internal standard and total PCBs as estimated ("UJ"). If extremely low sample area counts (<35% of the associated CCV or <25% of the associated ICV) are reported, qualify positive results for the homolog groups quantitated using that internal standard and total PCBs as estimated ("J") and qualify "not-detected" results for the homolog groups quantitated using that internal standard and total PCBs as unusable ("R"). If an internal standard RT varies by more than 10 seconds and no peaks are observed in the sample chromatogram, qualification of data is not necessary. Use professional judgment if peaks are observed in the sample chromatogram.

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Retention Time (RT) Windows (See Note #4 for additional information.)	The time (scan number) for initiation of data acquisition with each ion set must be carefully determined from the RTs (scan numbers) of the RT congeners in the PCB Window defining mixture. Approximate relative RTs of calibration congeners and approximate relative RT windows for PCB isomer groups are shown on Table 7 of SOP GEHR680. Absolute RTs of PCB congeners #77, #104, #202, and #189 should not vary by more than ±10 s from one analysis to the next of the PCB Window defining mixture. (RT reproducibility is not as critical for congeners #1 and #209 as for the other four congeners, which are used to determine when ion sets are changed.)	If the PCB Window defining mixture RTs are not within the specified RT windows, evaluate sample ion current profiles (ICPs) for false positives and false negatives. If a constant drift in RT is observed in the bracketing PCB Window defining mixtures, the direction of the RT drift should be applied to the sample ICPs.

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Blanks (See Note #5 and Note #10 for additional information.)	Summarize all results greater than the method detection limit (MDL) present in the blanks (identified by PCB congener). The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a PCB congener is found in the blank but not in the associated sample(s), no action is required. If a PCB congener in a sample is ≤5× the blank result, subtract the PCB congener concentration from the associated homolog group and total PCB results (and note in the validation report). If all PCB congeners of a homolog group result are ≤5× the blank results, qualify the positive result for the homolog group as "not detected" ("U*") and subtract the homolog group result from the total PCB result. If the positive result qualified "U*" is <RL, the RL should be used as reported. If the positive result qualified "U*" is ≥RL, the value of the positive result should be used as the revised RL. If a PCB congener in a sample is >5× blank result, no action/qualification is required. If gross contamination exists (*i.e.*, saturated peaks on the GC/MS), qualify the positive results as unusable ("R") due to interference.
Surrogates (See Note #6 for additional information.)	Use 60-140% as limits.	If the recoveries of one or more surrogates are > upper limit, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the recoveries of one or more surrogates are < lower limit but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recoveries of one or more surrogates are <10%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD) (**IF REQUESTED**)	**Calculated for Total PCBs only.** For accuracy, use recovery limits of 60-140%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if the original concentration of Total PCBs is >4× the spiking level for Total PCBs. RPDs calculated using MS/MSD results can be used to evaluate precision. If the recovery is >140%, qualify the positive total PCB results in the native sample as estimated ("J") and do not qualify the "not-detected" total PCB results. If the recovery is <60% but ≥10%, qualify the positive total PCB results in the native sample as estimated ("J") and qualify the "not-detected" total PCB results in the native sample as estimated ("UJ"). If the recovery is <10%, qualify the positive total PCB results in the native sample as estimated ("J") and qualify the "not-detected" total PCB results in the native sample as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive total PCB results in the native sample as estimated ("J") and do not qualify the "not-detected" total PCB results. If a field duplicate of the native sample was collected and analyzed, the field duplicate sample should also be qualified if an MS/MSD recovery or RPD is outside of criteria (as stated above for the native sample).
Laboratory Control Samples (LCS)	**Calculated for Total PCBs only.** For accuracy, use recovery limits of 60-140%.	If the recovery is >140%, qualify positive total PCB results in all associated samples as estimated ("J") and do not qualify "not-detected" total PCB results. If the recovery is <60% but ≥10%, qualify positive total PCB results in all associated samples as estimated ("J") and qualify "not-detected" total PCB results in all associated samples as estimated ("UJ"). If the recovery is <10%, qualify positive total PCB results in all associated samples as estimated ("J") and qualify "not-detected" total PCB results in all associated samples as unusable ("UR").

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Field/Laboratory Duplicate (See Note #7 and Note #10 for additional information)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are ≥5× RL. Use limit of ± RL (±2× RL for solids) when at least one sample value is <5× RL. (Use one-half the RL as a numerical value for any "not-detected" results in the RPD calculations). Compare both individual total homolog results and total PCB results separately.	If the criteria are not met, qualify positive results for the out-of-criteria total homolog group or total PCBs in the original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Percent Solids	Solid samples with less than 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.

Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Quantitation and Qualititative Identification (See Notes #4, #8, and #9 for additional information.)	Calculations should be performed in accordance with Section 12 of SOP GEHR680. Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution. EICPs must be evaluated to determine whether the laboratory correctly identified the PCB congeners based upon the identification procedures and criteria defined in Sections 11.3 and 11.4 of SOP GEHR680.	If a PCB congener result exceeds the instrument calibration range, qualify the positive result fro the associated homolog group as estimated ("J"). Use professional judgement to determine whether sample reanalyses and dilutions should be compared to the original analyses. If criteria (see field duplicate usability) between the original sample results and the reanalysis sample results are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement to determine whether qualititative identifications are accurate and whether data qualification is necessary.
System Performance (See Note #9 for additional information.)	Professional judgement should be used when assessing the degradation of system performance during analyses.	Use professional judgement to qualify the data if it is determined that system performance degraded during sample analyses.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitations of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

Notes for the Validation of PCB Data Generated by US EPA Method 680

1. Criteria for DFTPP Spectrum

m/z	Relative Abundance
127	40-60%
197	<1%
198	100% (Base Peak)
199	5-9%
275	10-30%
365	>1%
441	Present and <m/z 443
442	>40%
443	17-23% of m/z 442

2. If the initial calibration curve %RSD>50%, the linearity of the first three initial calibration standards should be evaluated. If the first three initial calibration standards for the PCB congener are linear (*i.e.*, r ≥0.99), do not qualify "not-detected" results. If the first three initial calibration standards for the PCB congener are not linear, qualify "not-detected" results as estimated ("UJ").

3. If instrument instability (*i.e.*, several CCV standards with PCB congeners exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential CCV standards, "not-detected" results may be qualified as estimated ("UJ") due to instrument sensitivity of a CCV standard response that is greater than the initial calibration standard response (increase in instrument sensitivity).

Notes for the Validation of PCB Data Generated by US EPA Method 680

If the CCV standard is %D>20% in the direction of increased instrument sensitivity and it is determined that "not-detected" results should not be qualified, the data reviewer should note this within the QAR support documentation.

4. Use professional judgement when evaluating sample ion current profiles (ICPs) when an RT shift is observed. If the ICPs reveal peaks corresponding to PCB congeners of interest using expanded RT windows and the surrogate compounds do not display a similar shift in RT, the concentrations of the PCB congeners that are outside of the RT window are subtracted from the associated total homolog and total PCB results.

   If the ICPs reveal peaks that interfere with potential detection of a PCB congener, qualify reported positive results for the associated total homolog group as unusable ("R").

5. The frequency of equipment/rinse blanks is determined during the sampling event. The results of a equipment/rinse blank should be applied to all samples collected in the same day, unless only one blank was collected for a several-day sampling event. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant.

   If a sample result qualified "U*" is <RL and the laboratory did not report the RL on the data tables or Form I, the positive result (*e.g.*, 8 µg/L) should be replaced with the RL (*e.g.*, 10 µg/L).

Notes for the Validation of PCB Data Generated by US EPA Method 680

Instrument blank contamination should be applied to samples bracketing the contaminated instrument blank.

6. The surrogate recovery limits do not apply to samples analyzed at greater than five-fold dilutions. Qualification of the data is not necessary if the surrogate is diluted beyond detection. Generally, a greater than five-fold dilution will affect the ability to even detect the surrogate. If a sample was analyzed at a five-fold dilution or less and either surrogate was not detected in the sample, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Write a comment in the QAR addressing the issue that sample-specific method performance based on surrogate recoveries could not be evaluated due to the dilution required for sample analysis.

7. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. Laboratory duplicate results and field duplicate results apply only to the original sample and the laboratory/field duplicate. Soil duplicate results are expected to have greater variance than aqueous duplicate results.

8. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

Notes for the Validation of PCB Data Generated by US EPA Method 680

9. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

- High background levels or shifts in absolute RTs of internal standards
- Excessive baseline rise at elevated temperature
- Extraneous peaks
- Loss of resolution
- Peak tailing or peak splitting that may result in inaccurate quantitation

10. The RL will be defined on a project-specific basis. If the project-required RL is less than the low calibration standard concentration, the Project Manager should be consulted for instructions about application of qualification related to the RL.

APPENDIX 38

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards, Inc. data reviewer will use to validate polychlorinated dibenzodioxin (dioxin) and polychlorinated dibenzofurans (furan) organic data generated by US EPA Method 1613B for General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to US EPA Method 1613B and/or other reference documents (*e.g.*, analytical SOPs), as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the dioxin/furan organic data provided by the analytical laboratory(ies) will be determined based on the general guidance provided in the "US EPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Validation" (Draft 9/2000) (National Functional Guidelines). It should be noted that the National Functional Guidelines applies strictly to data generated by the Contract Laboratory Program (CLP) protocol. As such, it is not directly applicable to validation of data generated by US EPA Method 1613B; therefore, this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared from one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and all data qualifier codes, or a data summary spreadsheet of the qualified analytical results will be included in the analytical results section of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELforms:

Organic field duplicate comparison Rev 1-01.xls
Organic field quadruplicate comparison Rev 1-01.xls
Organic field triplicate comparison Rev 1-01.xls

Chemistry Apps

FIT
Methods database

3.0 REFERENCE DOCUMENTS

US EPA Method 1613B (10/94)

US EPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Validation (Draft 9/2000)
Region III – SOP for Dioxin/Furan Data Validation (Draft 3/99)
Region IV – Data Validation SOP for Polychlorinated Dibenzodioxin and Polychlorinated Dibenzofurans Analysis by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (9/96)
Region II – Data Validation SOP for EPA Method 1613, Revision A (Revision 2 9/99)

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the dioxin/furan data based on an evaluation of information presented in the data package deliverables. Compliance to US EPA Method 1613B and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Organic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or any certain aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of deficiencies prior to the submittal of the QAR (if feasible and sanctioned by General Electric Company) at a minimum corrections necessary for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would take a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as

a result of such requests will be discussed in the comments subdivision of the QAR and included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the dioxin/furan organic data based on an evaluation of the information presented in the data package deliverables. The findings of the dioxin/furan organic data usability assessment will be described in terms of certain qualifications of the data that the project team should consider in order to best utilize the data. These qualifications will be presented in the Organic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with representative qualifier code(s) in the General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank qualification, common contaminants that were not qualified, unusable results ("R/UR"), estimated results ("J/UJ"), field duplicate comparison and a general qualifier for all results reported the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the dioxin/furan organic data and the resulting qualifications will be as stated in the attached Table for the Validation of Polychlorinated Dibenzo-*p*-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B. It should be noted that the Project Manager should be consulted when directed to use "professional judgement" in the attached table.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	0-4°C for aqueous <-10°C for solid and tissue	Due to the stability of PCDDs and PCDFs, there is no direct impact on data usability due to receipt temperatures outside the specified range.
Holding Time (See Note #1 for additional information.)	All matrices should be extracted within 30 days of sample collection and analyzed within 45 days of extraction.	If holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If holding time is grossly exceeded (>twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Chromatographic Resolution (Isomer Specificity Test Standard, See Note #2 for additional information)	Should be analyzed at the beginning of each 12-hour period of sample and standard analysis. The % valley between unlabeled 2,3,7,8-TCDD and all other unlabeled TCDD should be <25%. The RT of the first and last eluting isomers are used to establish the RT windows for each congener class of PCDD/PCDF compounds.	Use professional judgement if the Isomer Specificity Test Standard was not analyzed at the required frequency. If the % valley between unlabeled 2,3,7,8-TCDD and all other unlabeled TCDD is >25%, qualify positive results for 2,3,7,8-TCDD as estimated ("J").
Window Defining Mix (WDM)	Should be analyzed at the beginning of each 12-hour period of sample and standard analysis.	If frequency is not met, qualify positive results for total homologues as estimated ("J").
Instrument Performance-Mass Spectrometer Performance (PFK)	Should be analyzed at the beginning of each 12-hour period during which samples are to be analyzed and prior to the analysis of the initial and continuing calibration standards. A static resolving power of at least 10,000 (10% valley definition) should be demonstrated at appropriate masses before any analysis is performed and at the end of each 12-hour period.	Use professional judgement if the mass calibration was not performed at the required frequency or if the resolving power was less than 10,000.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Initial Calibration (See Note #3 for additional information)	Should be established with a minimum of 5 different concentration levels. The %RSD should be ≤20% for the 17 unlabeled standards and ≤35% for the labeled reference compounds. The relative ion abundance ratios should be within the limits specified in Note #3. The retention times of all target compounds, internal standards, and recovery standard should be within the windows established. The two monitored ions for each homologue should be present and should maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ions. The signal-to-noise (S/N) ratio for the GC signals present in the selected ion current profiles (SICPs) should be ≥ 10.	If the %RSD >20% but ≤90% (for unlabeled), qualify positive result as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the %RSD >90% (for unlabeled), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the relative ion abundance ratios for the two quantitation ions of the target compounds, internal standard, and/or recovery standards were not with the stated range, qualify positive results as unusable ("R"). Qualify positive results associated with the out of criteria ion abundance ratio internal standards and/or recovery standards. If the retention time of any target compound, internal standard, and/or recovery standard is not within the established retention time windows (±10 seconds of retention times in the WDM), qualify all data as unusable ("R/UR"). If the two monitored ions for a native isomer are not present and/or did not maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ion, qualify positive results as "not-detected" ("U") (the reported concentration with be reported at the detection limit). If the S/N ratio was <10, qualify "not-detected" results as unusable ("UR").

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Continuing Calibration (See Notes #3 and #4 for additional information)	Should be analyzed at the beginning of each 12-hour shift. The relative ion abundance ratios should be within the limits specified in Note #3. The recoveries (%Rs) should be within the limits specified in Note #4. The retention times for all compounds should be within the windows established. The two monitored ions for each homologue should be present and should maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ions. The signal-to-noise (S/N) ratio for the GC signals present in the SICPs should be ≥ 10.	If the unlabeled target compound recovery < lower limit but ≥ 50% of the lower limit, qualify positive result as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the unlabeled target compound recovery is > the upper limit but ≤ 150% of the upper limit, qualify positive result as estimated ("J"). If the unlabeled target compound recovery is < 50% of the lower limit or > 150% of the upper limit, qualify positive and "not-detected" results as unusable ("R/UR"). If the relative ion abundance ratio for the two quantitation ions are not within the specified range, qualify positive results as unusable ("R"). If the retention time of any target compound is not within the specified retention time window, qualify positive results as unusable ("R"). If the two monitored ions for a native isomer are not present and/or did not maximize simultaneously within 3 seconds of the corresponding ^{13}C-labeled isomer ion, qualify positive results as "not-detected" ("U") (the reported concentration with be reported at the detection limit). If the S/N ratio was <10, qualify "not-detected" results as unusable ("UR").
Internal Standards and Recovery Standards	Added to all samples and standards. %Rs should be within the limits specified in Note #4. The relative ion abundance ratios should be within the limits specified in Note #3. The retention times should be within the windows established.	Use professional judgement to determine if qualification is necessary due to relative ion abundance ratio being outside the specified range and if the retention times are not within the windows established. If the %R is > upper limit, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the %R is < lower limit but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the %R is <10%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Blanks (See Note #5 for additional information)	Summarize all results greater than the estimated detection limit (EDL). The highest positive results associated with a sample should be utilized for evaluation of contamination.	If a target compound is found in blank but not in the associated sample(s), no action is taken. If a sample result is ≤5× the blank result, qualify the results as "not-detected" ("U*"). The value of the positive result should be used as the revised EDL. If a sample result >5× (or 10× for OCDD only) blank result, no qualification is necessary. If gross contamination exists (*i.e.*, saturated peaks by GC/MS), qualify samples as unusable ("R") due to interference.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	For accuracy, use laboratory acceptance limits. For precision, use RPD limit of 40% for all matrices.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if original concentration of a compound is > 4× spiking level for that compound. RPDs calculated using MS/MSD results can still be used to evaluate precision. If the recovery is > upper limit, qualify positive results for that compound in the native sample as estimated ("J") and do not qualify "not-detected" results. If the recovery is < lower limit but ≥10%, qualify positive results for that compound in the native sample as estimated ("J") and qualify "not-detected" results for that compound in the native sample as estimated ("UJ"). If the recovery is <10%, qualify positive results in the native sample as estimated ("J") and qualify "not-detected" results for that compound in the native samples as unusable ("UR"). If the precision is >20%, qualify positive results for that compound in the native sample as estimated ("J") and do not qualify "not-detected" results. If the precision criteria (See field duplicate usability criteria) for non-spiked compounds are not met, qualify positive results in the native sample as estimated ("J") and qualify "not-detected" results in the native sample as estimated ("UJ"). If a field duplicate of the native (unspiked) sample was collected and analyzed, the field duplicate should also be qualified if the MS/MSD %Rs or RPD are outside of criteria as stated above for the native sample.
Ongoing Precision and Recovery (OPR) Standard	%Rs should be within the limits specified in Note #4. The relative ion abundance ratios should be within the limits specified in Note #3. The retention times should be within the windows established.	If the recovery for a target compound is outside of the acceptance criteria, qualify all positive results and "not-detected" results as unusable ("UR").

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Field Duplicate/Laboratory Duplicate (See Note #6 for additional information)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are ≥5× RL. Use limit of ± RL (±2× RL for solids) when at least one sample value is <5× RL. (Use one-half the RL as a numerical value for any "not-detected" results in the RPD calculations).	If the criteria are not met, qualify positive results in original sample, and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Percent Solids	Qualification is for solid samples with less than 50% solid content.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Target compound Identification (See Note #7 for additional information)	For 2,3,7,8-substitued isomers for which an isotopically labeled internal standard is present, the absolute RT at the maximum peak height should be within –1 to +3 seconds of the RT of the corresponding labeled standard. For non-2,3,7,8-substitued isomers, the RT should be within the established window. The two quantitation ions for the compounds, internal standards, and recovery standards should maximize simultaneously (within 2 seconds). The relative ion abundance ratios should be within the limits specified in Note #3. All integrated ion current for each characteristic ion of the target compound should have an S/N ratio ≥2.5. The identification of a peak as a PCDF can only be made if no signal having a S/N ≥2.5 is detected at the same time in the corresponding polychlorinated diphenyl ether (PCDPE) channel. Any results reported for 2,3,7,8-TCDF should be confirmed on a DB-225 column.	Use professional judgement to determine if the result should be changed to "not-detected" or flagged "EMPC" if one or more of the identification criteria specified was not met. Use professional judgement if a PCDPE peak was detected at the same retention time as a reported PCDF result.
Compound Quantitation and Detection Limits	The laboratory should reextract samples (utilizing a smaller sample aliquot) with compound concentrations above the instrument calibration range.	If a target compound result exceeds the instrument calibration range, qualify the positive result as estimated ("J"). If a target compound result is below the low calibration standard concentration, qualify the positive result as estimated ("J"). If the laboratory performed a dilution of a sample that had a target compound result that exceeded the instrument calibration range instead of reextracting a smaller sample aliquot, qualify positive results for the dilution analysis as estimated ("J").
System Performance (See Note #8 for additional information)	Professional judgement should be used when assessing the degradation of the system performance during analyses.	Professional judgement should be used to qualify the data if it is determined that the system performance has degraded during sample analysis.

Table for the Validation of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data Generated by US EPA Method 1613B

Quality Control Item	Usability Criteria	Action
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data that were not qualified based on the QC previously addressed. Write a brief narrative to give the user an indication of the analytical limitation of the data. If sufficient information on the intended use and required quality of the data is available, the reviewer should include the assessment of the usability of the data within the given context.

Notes for the Validation of PCDD and PCDF Data Generated by the US EPA Method 1613B

1. The holding time of extraction within 30 days of sample collection is a recommendation; however, since PCDDs and PCDFs are very stable in many matrices, the holding time may be as high as one year. Use professional judgement when evaluating samples that were extracted beyond the 30 day holding time.

2.

DB-5 Column GC Retention Time WDM

Congener	First Eluted	Last Eluted
TCDF	1,3,6,8-	1,2,8,9-
TCDD	1,3,6,8-	1,2,8,9-
PeCDF	1,3,4,6,8-	1,2,3,8,9-
PeCDD	1,2,4,7,9-	1,2,3,8,9-
HxCDF	1,2,3,4,6,8-	1,2,3,4,8,9-
HxCDD	1,2,4,6,7,9-	1,2,3,4,6,7-
HpCDF	1,2,3,4,6,7,8-	1,2,3,4,7,8,9-
HpCDD	1,2,3,4,6,7,9-	1,2,3,4,6,7,8-

DB-5 Column TCDD Isomer Specificity Test Standard

1,2,3,4-TCDD	1,2,3,7-TCDD
1,2,3,9-TCDD	2,3,7,8-TCDD

DB-225 Column TCDF Isomer Specificity Test Standard

2,3,4,7-TCDF
2,3,7,8-TCDF
1,2,3,9-TCDF

3. If the initial calibration %RSD is >50% but ≤90%, the linearity of the first three initial calibration standards for the compound should be evaluated. If the first three initial calibration standards for the compound are linear then do not qualify "not-detected"

results. If the first three initial calibration standards for the compound are not linear, then qualify "not-detected" results as estimated ("UJ").

Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive then the samples should be evaluated for false positives. If the concentration intercept is negative then the samples should be evaluated for false negatives.

Relative Ion Abundance Criteria for PCDDs and PCDFs

PCDDs	Relative Intensity
Tetra	0.65-0.89
Penta	1.32-1.78
Hexa	1.05-1.43
Hepta	0.88-1.20
Octa	0.76-1.02
PCDFs	Relative Intensity
Tetra	0.65-0.89
Penta	1.32-1.78
Hexa	1.05-1.43
Hexa[1]	0.43-0.59
Hepta	0.88-1.20
Hepta[2]	0.37-0.51
Octa	0.76-1.02

1 - used only for ^{13}C-HxCDF (internal standard)
2 - used only for ^{13}C-HpCDF (internal standard)

4.

Acceptance Criteria for Performance Tests When All PCDD/PCDF Are Tested

PCDD/PCDF	Concentration (ng/ml)	OPR (ng/ml)	Verification (ng/ml)
2,3,7,8-TCDD	10	6.7-15.8	7.8-12.9
2,3,7,8-TCDF	10	7.5-15.8	8.4-12.0
1,2,3,7,8-PeCDD	50	35-71	39-65
1,2,3,7,8-PeCDF	50	40-67	41-60
2,3,4,7,8-PeCDF	50	34-80	41.61
1,2,3,4,7,8-HxCDD	50	35-82	39-64
1,2,3,6,7,8-HxCDD	50	38-67	39-64
1,2,3,7,8,9-HxCDD	50	32-80	41-61
1,2,3,4,7,8-HxCDF	50	36-67	45-56
1,2,3,6,7,8-HxCDF	50	42-65	44-57
1,2,3,7,8,9-HxCDF	50	39-65	45-56
2,3,4,6,7,8-HxCDF	50	35-78	44-57
1,2,3,4,6,7,8-HpCDD	50	35-70	43-58
1,2,3,4,6,7,8-HpCDF	50	41-61	45-55
1,2,3,4,7,8,9-HpCDF	50	39-69	43-58
OCDD	100	78-144	79-126
OCDF	100	63-170	63-159
$^{13}C_{12}$-2,3,7,8-TCDD	100	20-175	82-121
$^{13}C_{12}$-2,3,7,8-TCDF	100	22-152	71-140
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	21-227	62-160
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	21-192	76-130
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	13-328	77-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	21-193	85-117
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	25-163	85-118
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	19-202	76-131
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	21-159	70-143
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	17-205	74-135
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	22-176	73-137
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	26-166	72-138
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	21-158	78-129
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	20-186	77-129
$^{13}C_{12}$-OCDD	200	26-397	96-415

Acceptance Criteria for Performance Tests When All PCDD/PCDF Are Tested

PCDD/PCDF	Concentration (ng/ml)	OPR (ng/ml)	Verification (ng/ml)
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.1-19.1	7.9-12.7

Acceptance Criteria for Performance Tests When Only Tetra Compounds Are Tested

PCDD/PCDF	Concentration (ng/ml)	OPR (ng/ml)	Verification (ng/ml)
2,3,7,8-TCDD	10	7.3-14.6	8.2-12.3
2,3,7,8-TCDF	10	8.0-14.7	8.6-11.6
$^{13}C_{12}$-2,3,7,8-TCDD	100	25-141	85-117
$^{13}C_{12}$-2,3,7,8-TCDF	100	26-126	76-131
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.7-15.8	8.3-12.1

Acceptance Criteria for Labeled Compound Recovery in Samples When All PCDD/PCDFs Are Tested

PCDD/PCDF	Concentration (ng/ml)	Recovery (ng/ml)	Recovery (%)
$^{13}C_{12}$-2,3,7,8-TCDD	100	25-164	25-164
$^{13}C_{12}$-2,3,7,8-TCDF	100	24-169	24-169
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25-181	25-181
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24-185	24-185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21-178	21-178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32-141	32-141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28-130	28-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26-152	26-152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26-123	26-123
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	29-147	29-147
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	28-136	28-136
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23-140	23-140
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28-143	28-143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26-138	26-138
$^{13}C_{12}$-OCDD	200	34-313	17-157
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.5-19.7	35-197

Acceptance Criteria for Labeled Compound Recovery in Samples When Only Tetra Compounds Are Tested

PCDD/PCDF	Concentration (ng/ml)	Recovery (ng/ml)	Recovery (%)
$^{13}C_{12}$-2,3,7,8-TCDD	100	31-137	31.137
$^{13}C_{12}$-2,3,7,8-TCDF	100	29-140	29-140
$^{37}Cl_4$-2,3,7,8-TCDD	10	4.2-16.4	42-164

5. The frequency of equipment blanks is determined during the sampling event. The results of a equipment/rinse blank should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day (unless only one was collected for a several-day sampling event; results would be applied to all samples in the SDG). In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration for a contaminant.

6. Duplicate samples may be taken and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. It is also expected that soil duplicate results will have a greater variance than aqueous duplicate results.

7. US EPA Method 1613B (Section 16.6) requires that a result meet all identification criteria or the result should not be reported. The sample should undergo reextraction with additional cleanup to remove any interference. Therefore, the laboratory should not be reporting the estimated maximum possible contamination (EMPC) results. If the presence of a reported positive is questioned (mostly due to chlorinated ether interference or if ratio/retention times are out), quality the result as "EMPC".

8. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

   a. high background levels or shifts in absolute retention times of internal standards
   b. excessive baseline rise at elevated temperatures
   c. extraneous peaks
   d. loss of resolution
   e. peak tailing or peak splitting that may result in inaccurate quantitation

APPENDIX 39

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate inorganic data generated by SW-846 Method 6010B for the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to SW-846 Method 6010B and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the inorganic data provided by the project laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory National Functional Guidelines for Inorganic Data Review" (2/94; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by SW-846 Method 6010B; this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes or a data summary spreadsheet of the qualified analytical results will be included in the analytical results section of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELForms:

- Inorganic field duplicate comparison Rev 1-01.xls
- Inorganic triplicate comparison Rev 1-01.xls
- Total versus dissolved comparison Rev 1-01.xls

Chemistry Applications:

- FIT
- Methods Database

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (2/94).

- SW-846 Method 6010B.

- Region I, EPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, Evaluation of Metals Data for the Contract Laboratory Program (CLP) (1/92) Validation of Inorganics.

- Region III, Modifications to Natural Functional Guidelines for Inorganic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the inorganic data based on evaluation of information presented in the data package deliverables. Compliance with SW-846 Method 6010B and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Inorganic Data Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of certain deficiencies prior to submittal of the QAR (if feasible and sanctioned by General Electric Company). At a minimum, corrections required to allow for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include

sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. In addition, the data reviewer should contact the project laboratories if feasible to request the correction of all correctable deficiencies that impact sample results or that the data reviewer was unable to correct prior to the submittal of the QAR, if time allows. Any laboratory resubmittals as a result of such requests will be discussed in the comments substation of the QAR and will be included as an attachment of the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the inorganic data based on an evaluation of the information presented in the data package deliverables. The findings of the inorganic data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data. These qualifications will be presented in the Inorganic Data Qualifier subsection of the QAR. Each qualification will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) in the General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination, unusable results (R/UR), estimated results (J/UJ), field duplicate comparison, and a general qualifier for all results reported below the quantitation/reporting limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the inorganic data and the resultant qualifications will be as stipulated on the attached Table for the Validation of Metals Data Generated by SW-846 Method 6010B. It should be noted that the Project Manager should be consulted when "professional judgement" use is indicated on the attached table.

Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Temperature and Conditions Upon Receipt	Aqueous samples should be preserved with nitric acid to pH≤2. Solid/soil samples should be preserved to 4±2°C.	If pH is >2 and the laboratory did not adjust the pH and allow the sample to sit for 16 hours before digestion, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Solid/soil samples should not be qualified due to out-of-criteria temperature upon receipt.
Technical Holding Time	All matrices should be analyzed within 6 months of sample collection.	If holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If holding time is grossly exceeded (>1 year from date of sample collection), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Initial Calibration	Should be established with a minimum of one blank and one standard.	Use professional judgement if the minimum number of standards was not used or if instrument was not calibrated daily and/or not calibrated each time set up.
Instrument Performance (See Note #1 for additional information.)	%D or %RSD between replicate exposures should be ≤20%. Samples should not display negative results >2× the instrument detection limit (IDL).	If %RSD or %D>20%, qualify positive results greater than the reporting limit as estimated ("J") and do not qualify "not-detected" results. If a negative result >5× IDL, qualify the "not-detected" result as unusable ("UR"). If an analyte displays a negative result >2× IDL, qualify the "not-detected" result as estimated ("UJ").
Initial Calibration Verification (ICV)	For accuracy, use recovery limits of 90-110%.	**Qualify samples for an entire analytical sequence.** If an analyte recovery is >110% but ≤125%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If an analyte recovery is <90% but ≥75%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If an analyte recovery is >125%, qualify positive results as unusable ("R") and do not qualify "not-detected" results. If an analyte recovery is <75%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Continuing Calibration Verification (CCV)	For accuracy, use recovery limits of 90-110%.	**Qualify samples analyzed before and after a non-compliant CCV.** If an analyte recovery is >110% but ≤125%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If an analyte recovery is <90% but ≥75%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If an analyte recovery is >125%, qualify positive results as unusable ("R") and do not qualify "not-detected" results. If an analyte recovery is <75%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
CRDL/CRI Standard (not required, but frequently analyzed.) (See Note #2 and Note #8 for additional information.)	For accuracy, use recovery limits of 85-115%.	**Qualify samples analyzed before and after a non-compliant CRDL/CRI standard.** If an analyte recovery is >115%, qualify positive results ≤3× the spike level as estimated ("J") and do not qualify "not-detected" results. If an analyte recovery is <85% but ≥50%, qualify positive results ≤ 3× the spike level as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If an analyte recovery is <50%, qualify positive results ≤3× the spike level as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If an analyte recovery is >150%, qualify positive results ≤ 3× the spike level as unusable ("R"), qualify positive results >3× the spike level but ≤5× as the spike level estimated ("J"), and do not qualify "not-detected" results.

Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Initial Calibration Blank (ICB)/Continuing Calibration Blank (CCB)/Preparation Blank (PB)/Field Blank/Equipment Blank (See Note #3 and Note #8 for additional information.)	The highest positive result (greater than the IDL) associated with a sample should be summarized and utilized for evaluation of contamination.	For ICBs and CCBs qualify samples per analytical sequence; for PBs, field blanks, and equipment blanks qualify per batch and for SDG. If an analyte is detected in the blank but not in the associated samples, no action is required. If a sample result is >MDL/DL but ≤5× blank result, qualify the positive result as "not-detected" ("U*"). If a sample result is >5× blank result, qualification is not required. If a blank has a negative result with an absolute value >2× IDL, qualify positive results ≤5× the absolute value of the blank result as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
ICP Interference Check Sample Analysis (ICSA/ICSAB) (See Note #4 for additional information.)	For accuracy, use recovery limits of 80-120% for ICSA/ICSAB. The absolute value of analytes not present in ICSA solution should be <2× IDL.	Qualify samples analyzed before and after ICSA/ICSAB standard. Sample data are acceptable if the concentrations of interferents (*i.e.*, Al, Ca, Fe, and Mg) in the samples are ≤ 50% of the respective ICSA concentrations. For samples with concentrations of interferents (*i.e.*, Al, Ca, Fe, and Mg) >50% of the respective concentrations in the ICSA, qualify as follows: If an ICSAB recovery is > 120%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If an ICSAB recovery is 50-79%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If an ICSAB recovery is <50%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If positive results are observed in the ICSA for non-ICSA analytes that are >2× DL, qualify positive results up to 5× ICSA concentration in samples with high (>50% ICSA interferents) interferents as estimated ("J") and do not qualify "not-detected" results. If negative results with an absolute value >2× DL are observed in the ICSA for non-ICSA analytes, qualify positive results up to 5× the concentration observed in the ICSA in samples with high (>50% ICSA interferents) interferents as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Laboratory Control Sample (LCS) (See Note #5 for additional information.)	For accuracy, use recovery limits of 80-120% for aqueous samples and 70-130% for solid samples.	For aqueous samples, if a recovery is >120% but ≤150%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. For aqueous samples, if a recovery is <80% but ≥50%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). For aqueous samples, if a recovery is >150%, qualify all positive results as unusable ("R") and do not qualify "not-detected" results. For aqueous samples, if a recovery is <50%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). For solid samples, if a recovery is >130%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. For solid samples, if a recovery is <70% but ≥30%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). For solid samples, if recovery is <30%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Matrix Spike/Matrix Spike Duplicates (MS/MSD) (See Note #6 and Note #8 for additional information.)	For accuracy, use recovery limits of 75-125%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated using %Rs) that are outside of criteria if the original concentration of an analyte is >4× the spiking level for that analyte. RPDs calculated using MS/MSD results can be used to evaluate precision. If a recovery is >125%, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If a recovery is <75% but ≥30%, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as estimated ("UJ"). If a recovery is <30%, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as unusable ("UR"). If the precision exceeds the RPD criterion, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results.
ICP Serial Dilution Analysis	%D<10% if original undiluted concentration is >50× IDL.	If %D is >10%, qualify positive results as estimated ("J") and do not qualify "not-detected" results.

Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Field Duplicate/Laboratory Duplicate (See Note #6, Note #7 and Note #8 for additional information.)	Use default limits of 20% RPD (%RSD for triplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate analyses) for solid samples when sample results are ≥5× RL. Use default limit of ± RL for aqueous samples and ±2× RL for solid samples when at least one sample result is <5× RL.	If the criteria are not met, qualify positive results for non-compliant analyte in original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Total vs. Dissolved Comparison (See Note #8 for additional information.)	When the dissolved result is greater than the total result: use default limits of ± IDL if at least one result is <10× IDL. Use default limit of percent difference <10% if both results are ≥10× IDL.	If the criteria are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If at least one result is <10× IDL and the difference is >5× IDL, qualify positive results and "not-detected" results as unusable ("R/UR"). If both results are ≥10× IDL and the percent difference is >50%, qualify positive results as unusable ("R").
Percent Solids	Solid samples with < 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitation of the data. If sufficient information of the intended use and required quality of the data is available, the reviewer should include the assessment of the usability of the data within the given content.

Notes for the Validation of Metals Data Generated by SW-846 Method 7470A/7471A

1. Due to the nature of trace ICP data, IDL may be very low such that 5× the IDL may be below the laboratory reporting limit. If the IDL is very low, use professional judgement to determine if the reporting limits should be qualified due to negative sample results.

2. If the spike concentration of the CRDL/CRI standard is < the IDL/MDL, do no utilize the results for qualification. Use professional judgement if the spike concentration of the CRDL/CRI standard is ≥ IDL but < the reporting limit.

3. Generally, if more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. When evaluating blank contamination, sample weights, volumes, and initial dilution factors should be taken into account. Sample results should not be blank corrected.

   The frequency of field/equipment/rinse blanks is determined during the sampling event. The results of a field/equipment/rinse blank should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day (if only one blank was collected for a several-day sampling event, results would be applied to all samples in the SDG).

4. When comparing ICSA results to sample results, the units of each should be the same (*i.e.*, if the sample results are in mg/kg and the ICSA results are in µg/L, convert the ICSA results to mg/kg before comparing the results.) If the negative interference in the ICSA solution is comparable (similar level) to the negative values observed in the CCBs,

Notes for the Validation of Metals Data Generated by SW-846 Method 7470A/7471A

the negative ICSA values should not be utilized for qualification. If the negative interference in the ICSA solution is not comparable to the negative values observed in the CCBs, the negative ICSA values should be utilized for qualification.

5. The spike level for the solid LCS should be comparable to the detection limit. Use professional judgement if the spike level is not comparable to the detection limit.

6. The laboratory may choose to analyze an MSD instead of a laboratory duplicate. The laboratory may include a post-digestion matrix spike (PDS) analysis. These results are not utilized for qualification; however, the results are utilized to evaluate the MS/MSD recoveries.

7. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. Field duplicate sample results should only be applied to the original sample and its field duplicate. Laboratory duplicates should be applied to all samples in a batch. It is also expected that soil duplicate results will have a greater variance than aqueous duplicate results.

8. The use of RL/DL in evaluating laboratory quality is as follows:

   - when evaluating negative values, total verses dissolved results, and the ICSA (non-spiked) compounds, the DL should be used.

Notes for the Validation of Metals Data Generated by SW-846 Method 7470A/7471A

- when evaluating field duplicates and laboratory duplicates, the RL/QL should be used.

The DL is defined as the number that the positive results are reported down to; therefore, the DL may be the IDL, MDL, or RL.

The RL is defined as the quantitation limit or project reporting limit. If the laboratory did not provide the RL, the IDL or MDL should be used.

APPENDIX 40

1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate mercury data generated by SW-846 Methods 7470A/7471A for General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to SW-846 Methods 7470A/7471A and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the mercury data provided by the analytical laboratory(ies) will be determined based on the general guidance provided in the "US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" (2/94; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by SW-846 Methods 7470A/7471A; this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared for one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes or a data summary spreadsheet of the qualified analytical results will be included in the analytical results section of the QAR.

2.0 EVALUATION TOOLS

Excel form available in R:/Templates/Chemistry/XCELForms:

- Inorganic field duplicate comparison Rev 1-01.xls
- Inorganic triplicate comparison Rev 1-01.xls
- Total versus dissolved comparison Rev 1-01.xls

Chemistry Applications:

- FIT
- Methods Database

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (2/94).
- SW-846 Methods 7470A/7471A.

- Region I, EPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, Standard Operating Procedure for the Validation of Inorganic Data Acquired Using SW-846 Methods 7470A/7471A (Rev. 2, 12/94).

- Region III, Modifications to National Functional Guidelines for Inorganic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the mercury data based on evaluation of information presented in the data package deliverables. Compliance with SW-846 Methods 7470A/7471A and/or other reference documents (*e.g.*, analytical SOPs) as applicable to the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Inorganic Data Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of deficiencies prior to submittal of the QAR (if feasible and sanctioned by the General Electric Company). At a minimum, corrections required to allow for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would require a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such request will be discussed in the comments subsection of the QAR and will be included as an attachment of the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the mercury data based on an evaluation of the information presented in the data package deliverables. The findings of the mercury data usability assessment will be presented in terms of data qualifications that the project team should consider in order to best utilize the data. These qualifications will be presented in the Inorganic Data Qualifier Section of the QAR. Each qualification will indicate that the affected sample result(s) has been flagged with representative qualifier code(s) in the General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination, unusable results (R/UR), estimated results (J/UJ), field duplicate comparison, and a general qualifier for all results reported below the quantitation/reporting limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the mercury data and the resultant qualifications will be as stipulated on the attached Table for the Validation of Mercury Data Generated by SW-846 Methods 7470A/7471A. It should be noted that the Project Manager should be consulted when "professional judgement" use is indicated on the attached table.

Table for the Validation of Mercury Data Generated by SW-846 Methods 7470A/7471A

Quality Control Item	Usability Criteria	Action(s)
Temperature and Conditions Upon Receipt	Aqueous samples should be preserved to pH ≤2 with HNO_3. Solid/soil samples should be preserved to 4±2°C.	If pH is >2 and the laboratory did not adjust the pH and allow the sample to sit for 16 hours before digestion, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Solid/soil samples should not be qualified due to out-of-criteria temperatures.
Technical Holding Time	All matrices should be analyzed within 28 days of sample collection.	If holding time is exceeded, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If holding time is grossly exceeded *i.e.*, twice the holding time), qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Initial Calibration (See Note #1 for additional information.)	Calibration should be daily and each time the instrument is set up, with at least one blank and three standards. r (linear) should be ≥ 0.995.	Use professional judgement if the appropriate number of standards is not used or if the instrument was not calibrated daily and/or not calibrated each time set up. If the r is <0.995 but ≥0.850, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If r is <0.850, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Instrument Performance	Samples should not display negative results with an absolute value >2× the instrument detection limit (IDL).	If a negative result with an absolute value >2× IDL is observed, qualify the "not-detected" result as estimated ("UJ").

Table for the Validation of Mercury Data Generated by SW-846 Methods 7470A/7471A

Quality Control Item	Usability Criteria	Action(s)
Initial Calibration Verification (ICV)	For accuracy, use recovery limits of 80-120%.	Qualify samples for an entire analytical sequence. If the recovery is >120% but ≤135%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the recovery is <80% but ≥65%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recovery is <65%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the recovery is >135%, qualify positive results as unusable ("R") and do not qualify "not-detected" results.
Continuing Calibration Verification (CCV)	For accuracy, use recovery limits of 80-120%.	Qualify samples analyzed before and after a non-compliant CCV. If the recovery is >120% but ≤135%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the recovery is <80% but ≥65%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recovery is <65%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the recovery is >135%, qualify positive results as unusable ("R") and do not qualify "not-detected" results.

Table for the Validation of Mercury Data Generated by SW-846 Methods 7470A/7471A

Quality Control Item	Usability Criteria	Action(s)
CRDL/CRA Detection Limit (DL) standard (not required but frequently analyzed.) (See Note #2 and Note #7 for additional information.)	For accuracy, use recovery limits 75-125%.	Qualify samples analyzed before and after a non-compliant CRDL/CRA standard. If the recovery is >125% but ≤150%, qualify positive results ≤ 2 × the spike level as estimated ("J") and do not qualify "not-detected" results. If the recovery is <75% but ≥ 50%, qualify positive results ≤ 2 × the spike level as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the recovery is <50%, qualify positive results ≤ 2 × the spike level as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the recovery is >150%, qualify positive results ≤ 2 × the spike level as unusable ("R"), qualify positive results >2× the spike level but ≤5× the spike level as estimated ("J"), and do not qualify "not-detected" results.
Initial Calibration Blank (ICB)/ Continuing Calibration Blank (CCB)/ Preparation Blank (PB)/Field Blank/Equipment Blank (See Note #3 and Note #7 for additional information.)	The highest positive result (greater than the IDL) associated with a sample should be summarized and utilized for the evaluation of contamination.	For ICBs and CCBs, qualify samples per analytical sequence; for PBs, field blanks, and equipment blanks, qualify per batch and/or SDG. If mercury is detected in blank but not in sample, no action is required. If a sample result is >MDL/IDL but ≤5× blank result, qualify the positive result as "not-detected" ("U*"). If sample is >5× blank result, qualification is not required. If a blank has a negative result with an absolute value >2 × IDL, qualify positive results ≤ 5 × the absolute value of the blank result as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

Table for the Validation of Mercury Data Generated by SW-846 Methods 7470A/7471A

Quality Control Item	Usability Criteria	Action(s)
Laboratory Control Sample (LCS) (See Note #4 for additional information.)	For accuracy, use recovery limits of 80-120% for aqueous samples and 70-130% for solid samples.	For aqueous samples, if the recovery is >120% but ≤150%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. For aqueous samples, if the recovery is <80%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). For aqueous samples, if the recovery is >150%, qualify positive results as unusable ("R") and do not qualify "not-detected" results. For aqueous samples, if the recovery is <50%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). For solid samples, if the recovery is >130%, qualify positive results as estimated ("J") and do not qualify "not-detected" results. For solid samples, if the recovery is <70% but ≥30%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). For solid samples, if the recovery is <30%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Table for the Validation of Mercury Data Generated by SW-846 Methods 7470A/7471A

Quality Control Item	Usability Criteria	Action(s)
Matrix Spike/Matrix Spike Duplicates (MS/MSD) (See Note #5 for additional information.)	For accuracy use recovery limits of 75-125%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated using %Rs) that are outside of criteria if the original concentration of an analyte is >4× the spiking level for that analyte. RPDs calculated using MS/MSD results can be used to evaluate precision. If the recovery is >125%, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery is <75% but ≥30%, qualify all positive results in all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as estimated ("UJ"). If the recovery is <30%, qualify positive results in all associated samples as estimated ("J") and qualify all "not-detected" results in all associated samples as unusable ("UR"). If the precision exceeds the RPD criterion, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results.
Field Duplicate/Laboratory Duplicate (See Note #6 and Note #7 for additional information.)	Use default limits of 20% RPD (%RSD for triplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate analyses) for solids when sample results are ≥5× DL. Use default limit of ±DL for aqueous samples and ±2× DL for solid samples when at least one sample result is <5× DL.	If the criteria are not met, qualify positive results for the non-compliant analyte in original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").

Table for the Validation of Mercury Data Generated by SW-846 Methods 7470A/7471A

Quality Control Item	Usability Criteria	Action(s)
Total vs. Dissolved Comparisons (See Note #7 for additional information.)	When the dissolved result is greater than the total result: use default limits of ± IDL when at least one result is <10× IDL. Use default limits of percent differences <10% when both results are ≥ 10× IDL.	If the criteria are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If at least one result is <10× IDL and the differences is >5× IDL, qualify positive and "not-detected" results as unusable ("R/UR"). If both results are ≥10× IDL and the percent difference is >50%, qualify positive results as unusable ("R").
Percent Solids	Solid samples with less than 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the nature of the analytical problems.	Use professional judgement to determine the need to qualify data that were not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitation of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

Notes for the Validation of Mercury Data Generated by SW-846 Method 7470A/7471A

1. Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive, samples should be evaluated for false positives. If the concentration intercept is negative, samples should be evaluated for false negatives. Furthermore, samples should not display negative values >2× the detection limit (DL).

   The laboratory may utilize a non-linear regression curve fit. Due to different software programs, it may not be possible to reproduce the laboratory results.

2. The action limit of 2× the spike level assumes that the spike level is based on the RL; therefore, if the RL is < the action limit, the CRDL/CRA standard results should be utilized to qualify the sample data. If the RL is > the action level, the CRDL/CRA standard results should not be utilized to qualify sample data.

3. Generally, if more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. When evaluating blank contamination, sample weights, volumes, and initial dilution factors should be taken into account. Sample results should not be blank corrected.

   The frequency of equipment blanks is determined during the sampling event. The results of a equipment/rinse blank should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day (if only one blank was

Notes for the Validation of Mercury Data Generated by SW-846 Method 7470A/7471A

collected for a several-day sampling event; results would be applied to all samples in the SDG).

4. The spike level for the solid LCS should be comparable to the detection limit. Use professional judgement if the spike level is not comparable to the detection limit.

5. The laboratory may choose to analyze an matrix spike duplicate instead of a laboratory duplicate.

6. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates that measure only laboratory performance. Field duplicate sample results should only be applied to the original sample and its field duplicate. Laboratory duplicate results should be applied to all samples in a batch. It is expected that soil duplicate results will have a greater variance than aqueous duplicate results.

7. The use of RL/DL in evaluating laboratory quality is as follows:

   - When evaluating negative values and total verses dissolved results, the DL should be used.
   - When evaluating field duplicates and laboratory duplicates, the RL/QL should be used.

Notes for the Validation of Mercury Data Generated by SW-846 Method 7470A/7471A

The DL is defined as the number that the positive results are reported down to; therefore, the DL may be the IDL, MDL, or RL.

The RL is defined as the quantitation limit or project-reporting limit. If the laboratory did not provide the RL then the IDL or MDL should be used.